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Y-817

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UNITED STATES ATOMIC ENERGY COMMISSION

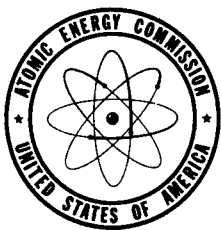
PRODUCTION OF ZIRCONIUM AT Y-12

By  
J. W. Ramsey  
W. K. Whitson, Jr.

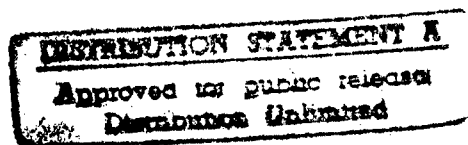
DTIC QUALITY INSPECTED 2

October 12, 1951

Carbide and Carbon Chemicals Company  
Oak Ridge, Tennessee



Technical Information Service, Oak Ridge, Tennessee



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Y-817

CARBIDE AND CARBON CHEMICALS COMPANY  
A DIVISION OF UNION CARBIDE AND CARBON CORPORATION

Y-12 PLANT

W-7405-Eng-26

CHEMICAL DIVISION

Mr. J. M. Herndon, Superintendent

CHEMICAL DEPARTMENT

Mr. G. A. Strasser, Superintendent

PRODUCTION OF ZIRCONIUM AT Y-12

J. W. Ramsey  
W. K. Whitson, Jr.

ABSTRACT

A general description is given of the permanent zirconium plant at Y-12. Equipment is described and materials of construction are listed. Photographs illustrating principal equipment and reduced construction drawings are also presented. Operating conditions and costs information are listed.

Oak Ridge, Tennessee

October 12, 1951

## INTRODUCTION

Production of purified hafnium-free zirconium was begun at Y-12 in January, 1950. At the request of the Atomic Energy Commission, a quick installation of equipment was made in order to produce 25,000 pounds of zirconium as oxide for initial experiments for the Naval Reactor Program. Less than 0.1% contained hafnium was specified. At that time, a program was started on designing a more efficient plant for the production of 150,000-200,000 pounds of zirconium per year. The permanent zirconium plant was completed in October, 1951. Additions were made to the extraction facilities and equipment for continuous purification by the phthalate process and continuous drying and calcining were provided.

At the time of this writing, the permanent zirconium plant is in the start-up stage. This report describes the equipment and process as they now exist and the operational plans which have been developed from experience and from laboratory and pilot plant work.

The original proposal for the permanent zirconium plant is outlined in a report, Y-573, "Separation of Zirconium and Hafnium - Proposal for Construction and Operation of Zirconium Production Plant", J. M. Googin and G. A. Strasser, March 14, 1950. These plans have been followed to completion with but few changes. Greater length of extraction and stripping columns was installed than was first planned in order to effect more complete separation

which was later requested. Later information obtained on calcining showed that protection against contamination in this stage was more difficult than had been expected, and consequently the expense of more elaborate calcination equipment was required. Corrosion of exteriors from vapors in the processing areas was found to be a serious problem and more elaborate ventilation and protective measures were taken than had been planned in the proposal. Otherwise the original proposal has been followed through approximately as first outlined.

It is suggested that reference should be made to report Y-573 relative to studying the report presented here.

### DESCRIPTION OF PROCESS

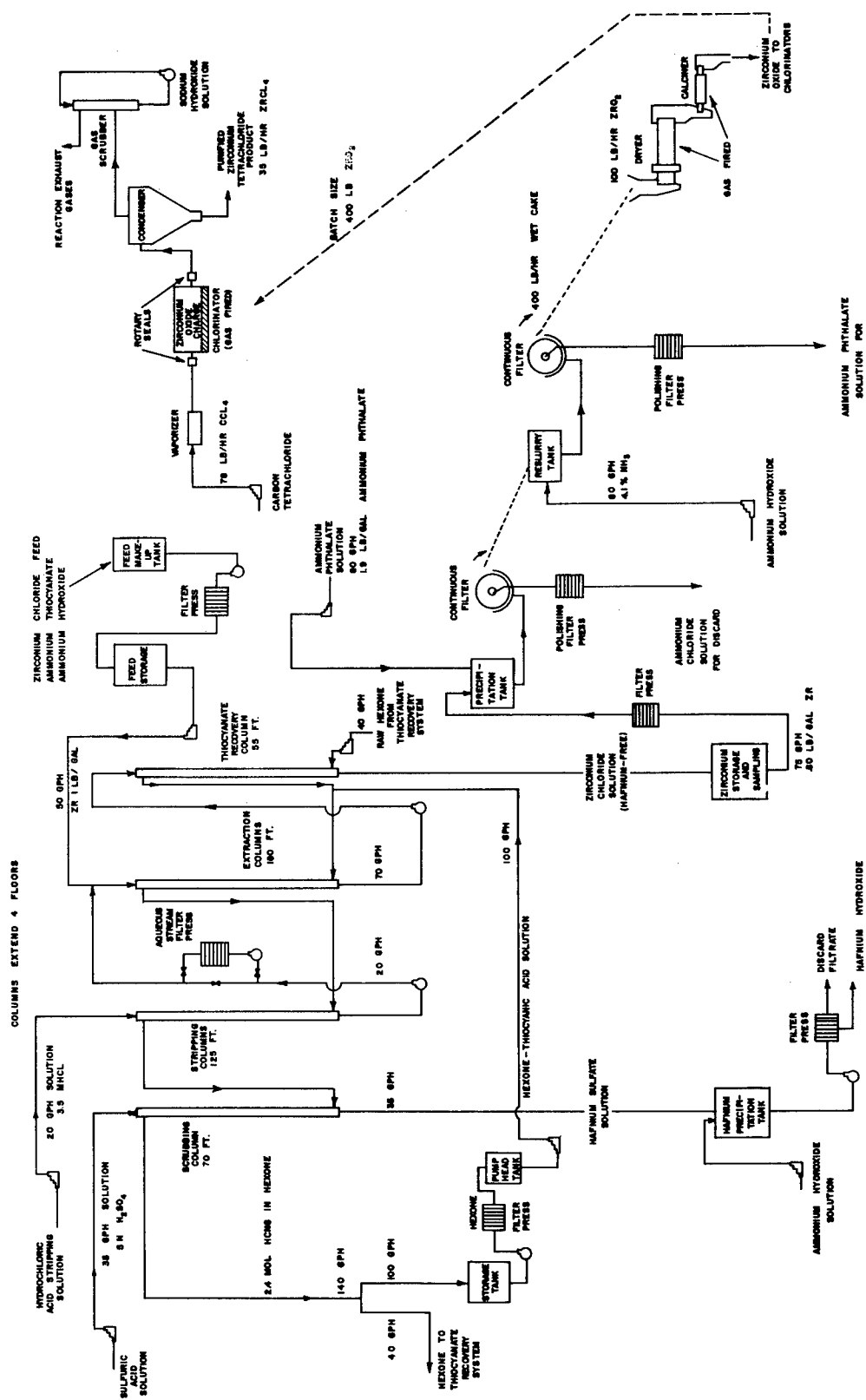
The attached flow sheet and photographs illustrate the permanent zirconium plant in Building 9211 at Y-12.

Zirconium tetrachloride, normally containing from 1.5 percent to 2.0 percent hafnium, is received from Titanium Alloy Manufacturing Division of the National Lead Company for use as feed material. Hafnium is removed from zirconium by an extraction process and resulting solutions are further purified by phthalate precipitation. Zirconium phthalate is converted to zirconium hydroxide by ammonium hydroxide leaching and the zirconium hydroxide is dried and calcined. The zirconium oxide is then chlorinated to form zirconium tetrachloride, which is used in magnesium reduction to the metal.

The steps in processing at Carbide and Carbon Chemicals Company, Y-12 Plant, are shown on the attached flow sheet and outlined as follows:

#### Hafnium Separation

Hafnium is separated from zirconium by a solvent extraction process employing methyl iso-butyl ketone. The separation is carried out in continuous counter-current spray towers. Solution containing normal zirconium is fed in the center of the extraction plant. The zirconium solution flows out the bottom of the plant while the hafnium is carried by the solvent to the top of the plant.



Zirconium tetrachloride is dissolved in water (top center of flow sheet) and the required quantities of ammonium thiocyanate and ammonium hydroxide are added to form the extraction feed solution. Some of the equipment used is shown in Figure 1, "Feed Make-up and Storage Area - Tank Pit". Feed solution is pumped to the column (Figure 2, "Base of Extraction Columns-First Floor"). There are three columns for extraction, two columns for stripping, one column for scrubbing, and one column for thiocyanate recovery. Columns are controlled by operators on the third floor (Figure 3, "Extraction Control Area").

Hafnium thiocyanate is preferentially extracted into hexone-thiocyanic acid solution, which is pumped into the bottom of the extraction column. Hexone from the extraction column flows into the stripping column, counter-current to a stripping solution of dilute hydrochloric acid. Aqueous stripper solution containing stripped zirconium is fed back into the extraction column with the extraction feed solution. Stripped hexone containing very pure hafnium flows into the scrubbing column where it is scrubbed with sulfuric acid solution. This hexone, free of metal, but still containing thiocyanic acid is recirculated to the extraction columns.

For smallest useage of thiocyanate, it is desirable to have thiocyanate concentration in the product stream at a very low level. This is accomplished



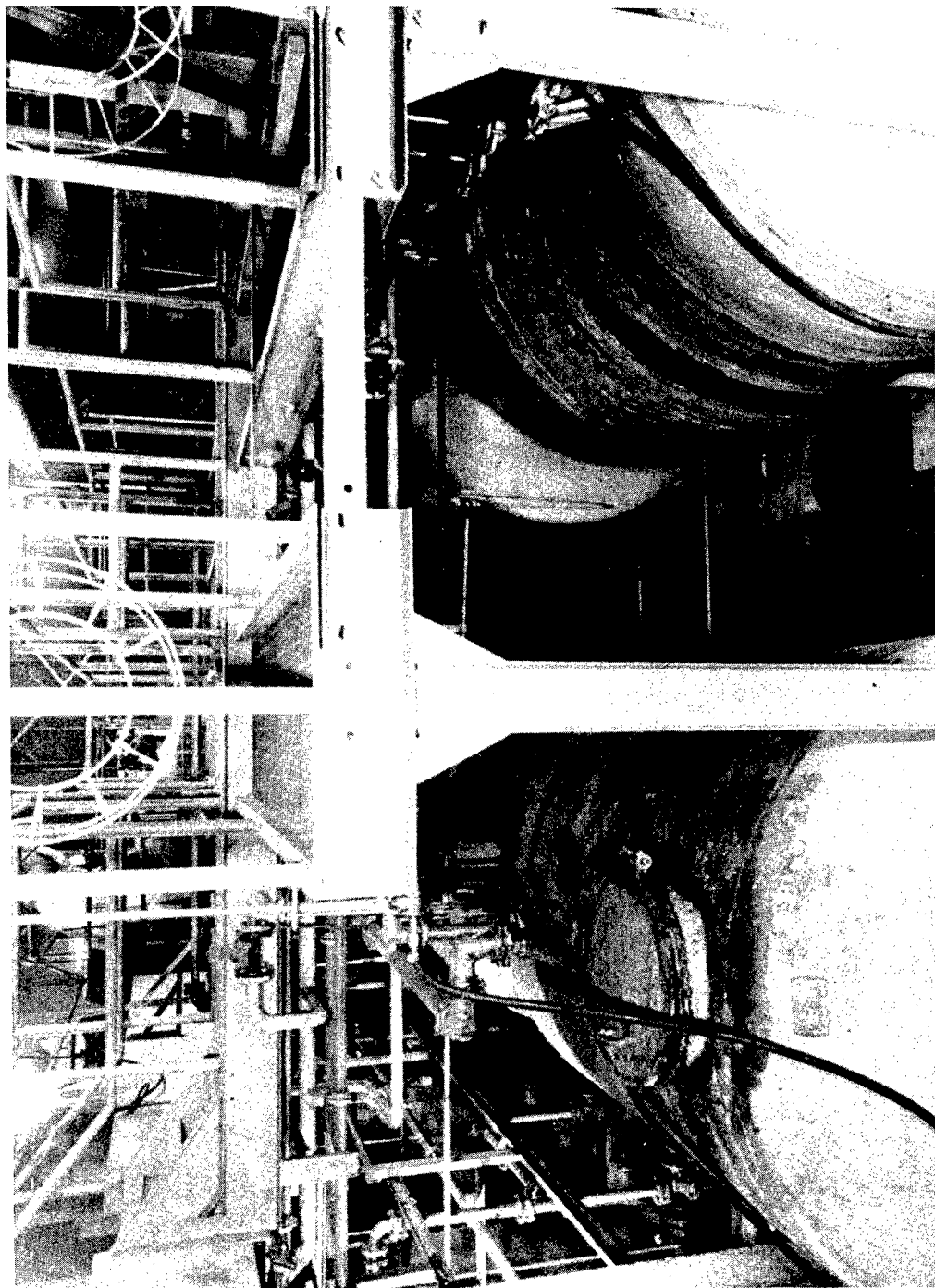


FIGURE 1. FEED MAKEUP AND STORAGE AREA -TANK PIT

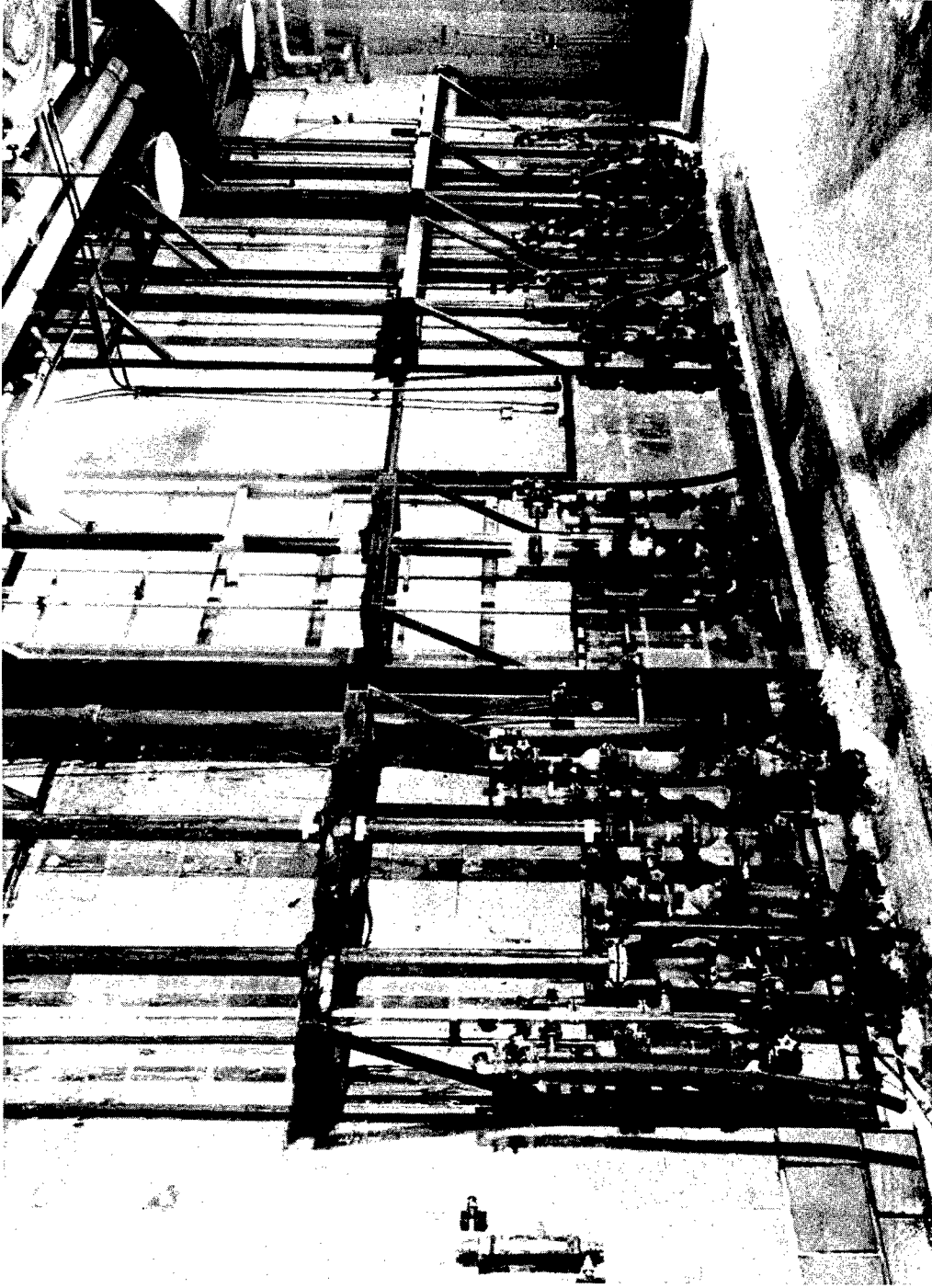


FIGURE 2. BASE OF EXTRACTION COLUMNS -- FIRST FLOOR

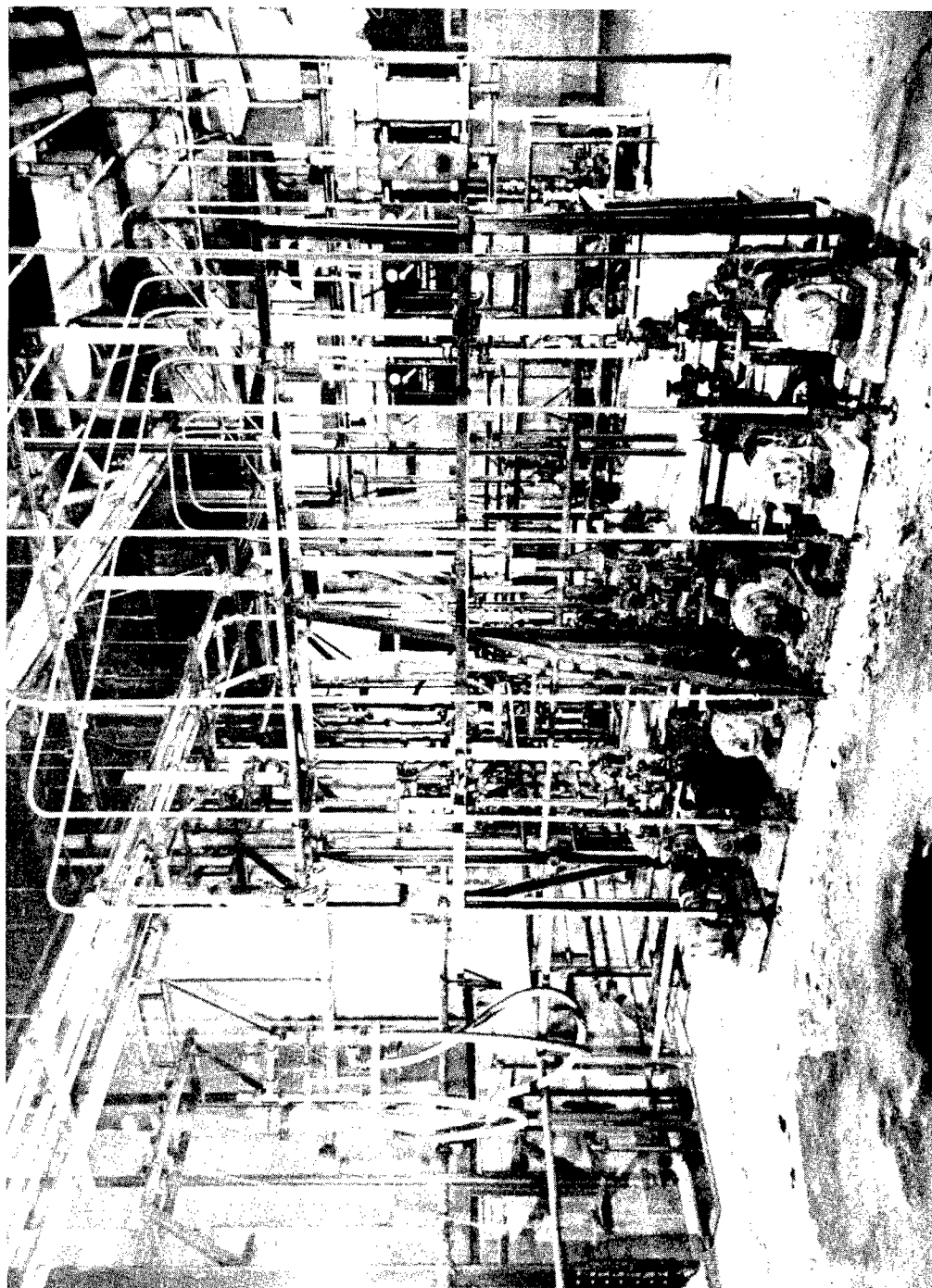


FIGURE 3. EXTRACTION CONTROL AREA - THIRD FLOOR

by directing the aqueous solution from the third extraction column into a thiocyanate recovery column. The thiocyanate recovery column is fed with raw hexone at a rate of approximately one-third the total hexone flow. Hexone from this column contains thiocyanic acid at the proper concentration for extraction and is mixed with the hexone entering the extraction columns. Raw hexone to be fed to the thiocyanate recovery column is prepared from a portion of the scrubbed hexone diverted to an ammonium neutralization system. Ammonium thiocyanate from this system is used in feed makeup.

Zirconyl chloride solution, hafnium-free, goes from the last extraction column to a tank for storage and sampling, and then to be further processed by precipitation with ammonium phthalate solution.

The hafnium is recovered from the hexone by sulfuric acid scrubbing. Hafnium is recovered from the sulfuric acid solution as hafnium hydroxide by precipitation with ammonium hydroxide.

#### Separation of Other Impurities

While hafnium is the element requiring special separation procedures, it is also necessary to remove other metal ions present as impurities in the feed material. This purification is carried out by precipitating zirconium as zirconyl phthalate. The phthalate precipitation is very selective for zir-

conium and hafnium, while other impurities, such as iron, copper, cadmium, etc., remain in solution and are thus separated.

In the permanent zirconium plant, ammonium phthalate solution and zirconium chloride solution are fed continuously to a precipitation tank, which, in turn, feeds a continuous Eimco filter. This equipment is shown in Figure 4, "Phthalate Precipitation Equipment and Filters." Cake is scraped continuously from the filter and reslurried with ammonium hydroxide solution. This slurry is filtered on a continuous Oliver filter. The ammonium phthalate solution from the filter is recovered by evaporation. (Figure 5, "Ammonium Phthalate Evaporator").

Zirconium hydroxide cake from the Oliver filter falls from the filter scraper blade through a chute into a continuous gas-fired drier, manufactured by the Bartlett-Snow Company. This is shown in Figure 6, "Assembly Work on Drier - Third Floor." The dried zirconium hydroxide falls continuously into silica-lined calciners in which it is converted to high purity zirconium oxide, (Figure 7, "Calciner - Second Floor"). Calciners were also manufactured by the Bartlett-Snow Company, and liners are supplied by the Amersil Company and the General Ceramics Company.

Hafnium hydroxide is redissolved and purified by the same chemical process

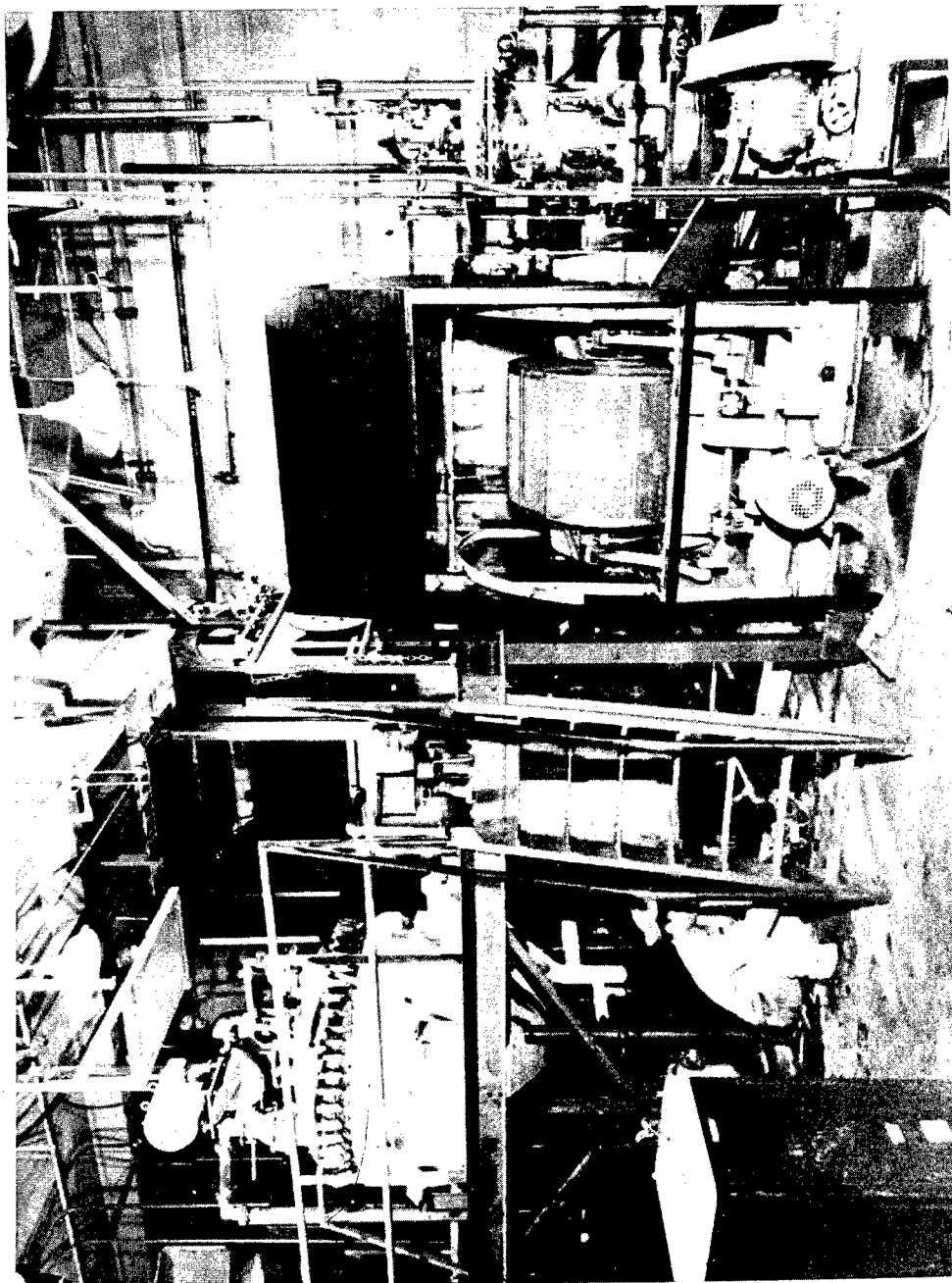


FIGURE 4. PHTHALATE PRECIPITATION EQUIPMENT AND FILTERS -  
FOURTH FLOOR

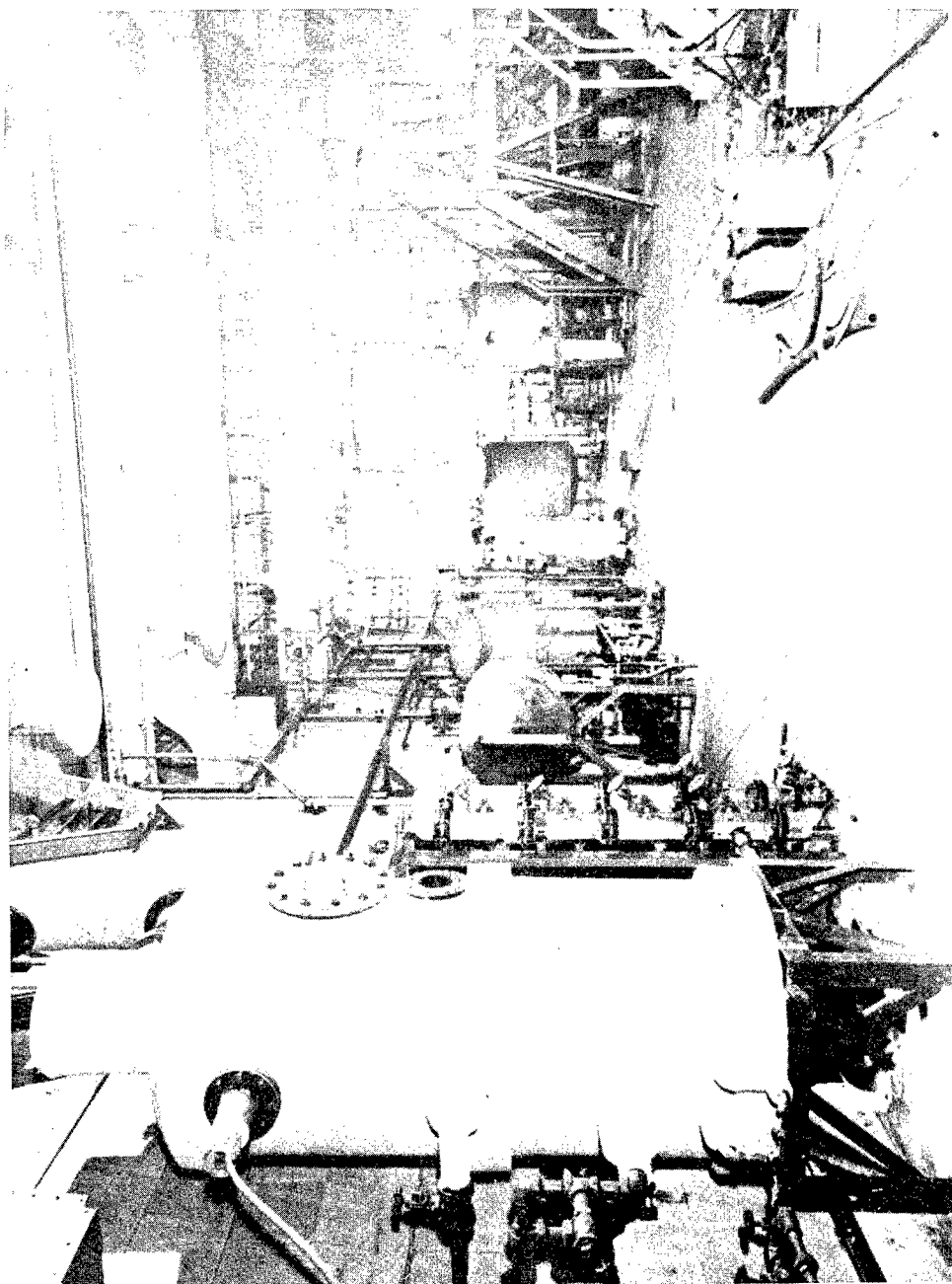


FIGURE 5. AMMONIUM PHTHALATE EVAPORATOR, MISCELLANEOUS HEAD  
TANKS IN BACKGROUND - FOURTH FLOOR

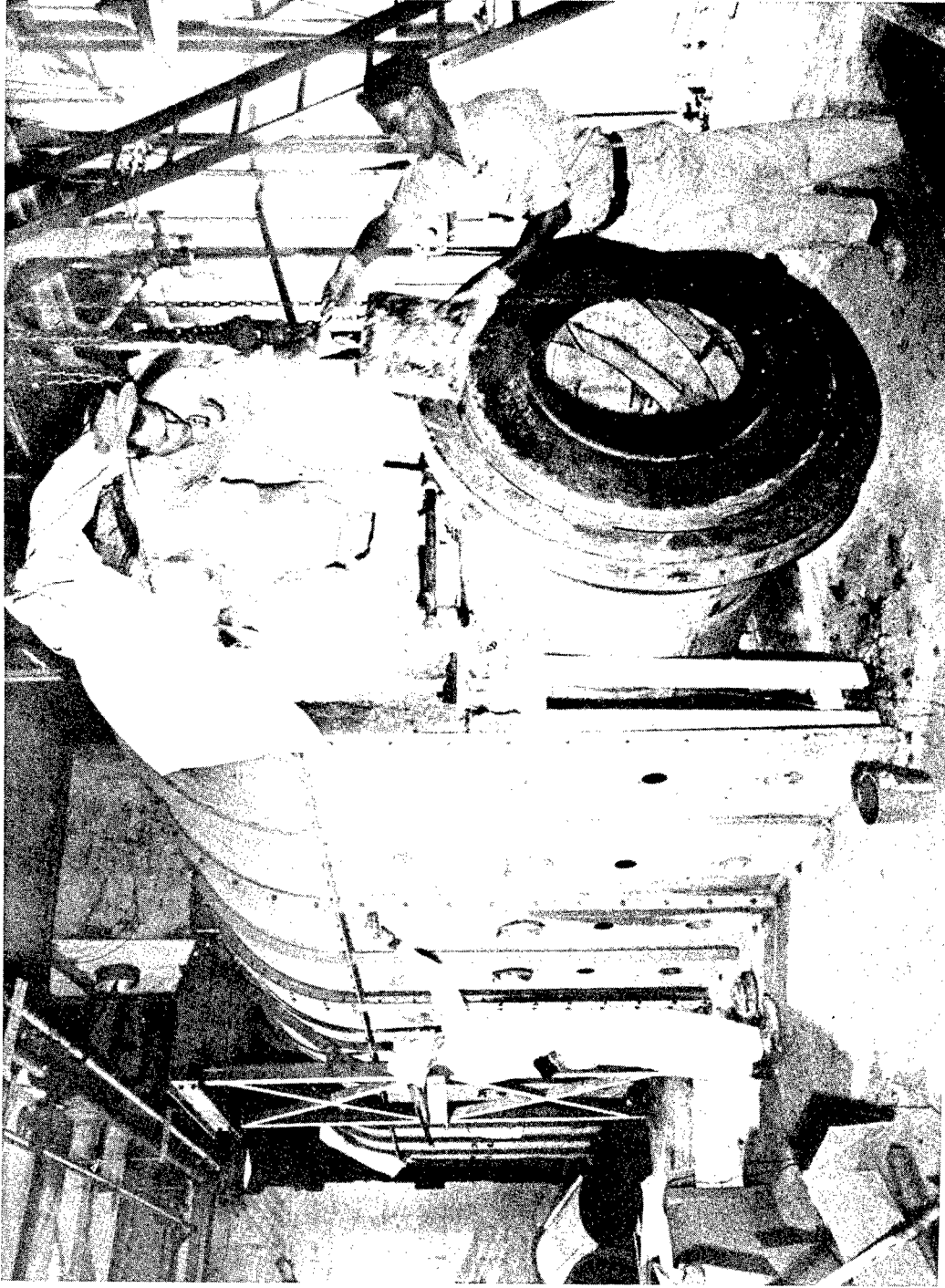


FIGURE 6. ASSEMBLY WORK ON DRIER - THIRD FLOOR



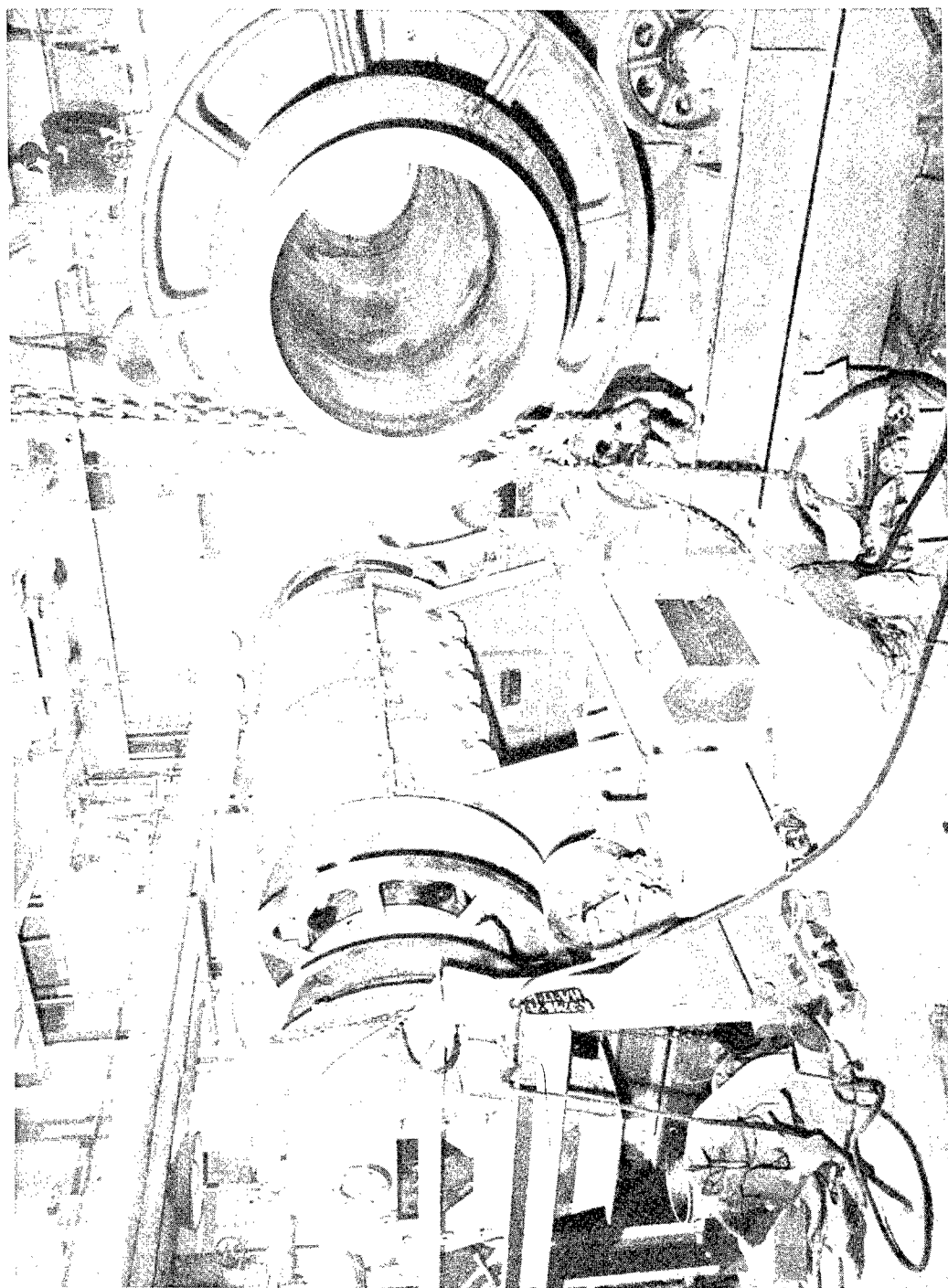


FIGURE 7. CALCINERS - SECOND FLOOR.

used for zirconium hydroxide. Some of the equipment in which this work is carried out may be seen in Figure 8, "Hafnium Purification Equipment."

### Chlorination

The method of chlorination that was used at Y-12 consisted of direct chlorination of the oxide with carbon tetrachloride in a rotary horizontal reactor. Zirconium oxide was charged batchwise into the reactor. Carbon tetrachloride was fed through a vaporizer into the rotary reactor forming volatile zirconium tetrachloride. The zirconium tetrachloride gas was collected in an air-cooled condenser and cleaned batchwise into shipping containers. The reaction gases were scrubbed in a sodium hydroxide system, (Figure 9, "Control Panel and Condensers of Horizontal Chlorinators - First Floor").

## MATERIALS OF CONSTRUCTION

### Handling of Process Materials

General selection of materials of construction at various stages of processing is given in Table I, "Materials of Construction for Handling of Process Materials". This table gives the actual construction of the permanent zirconium plant. Selection has been made based on chemical resistance to process solutions of various materials, and availability of standby equipment at Y-12.

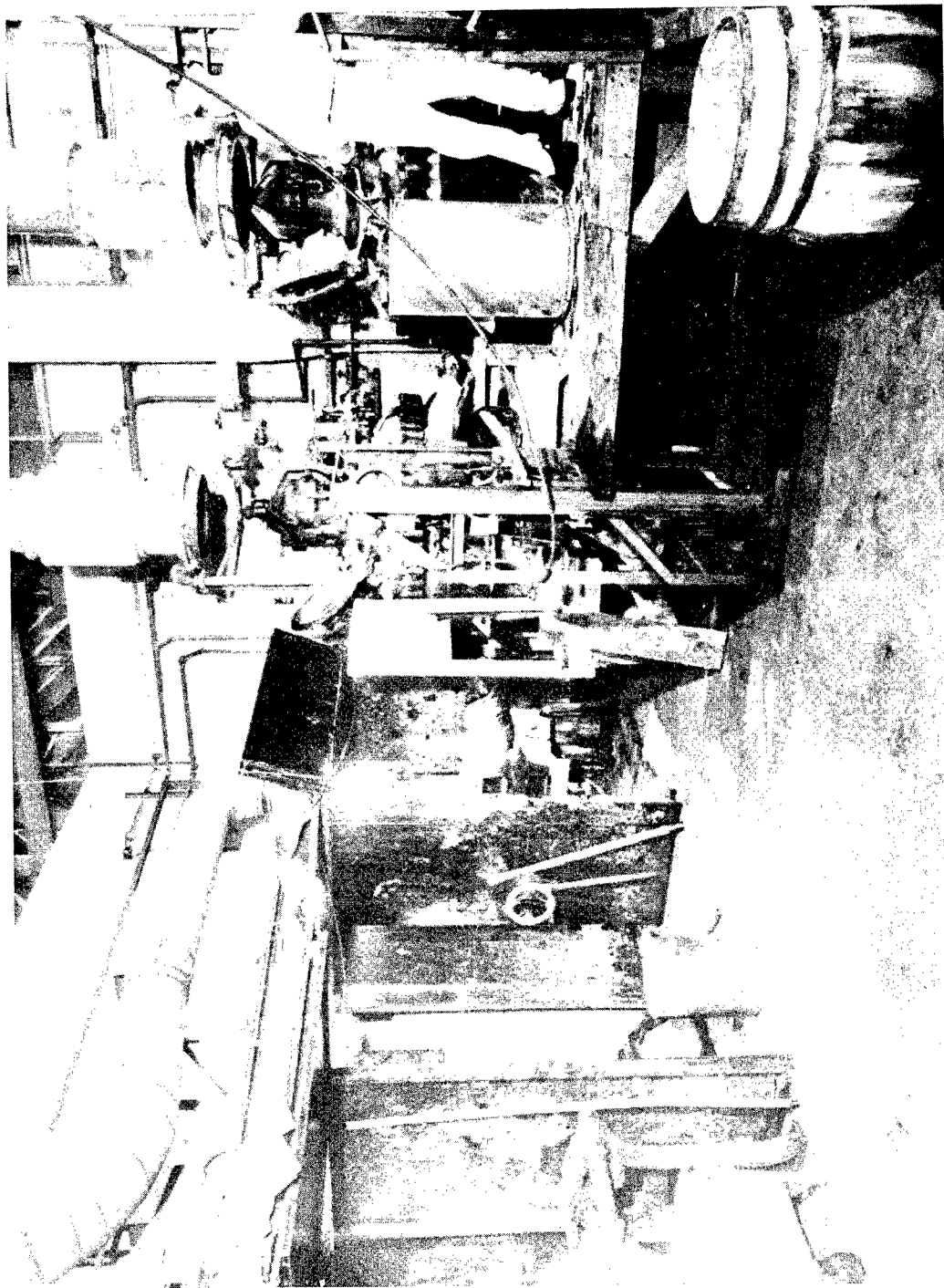


FIGURE 8. HAFNIUM PURIFICATION EQUIPMENT

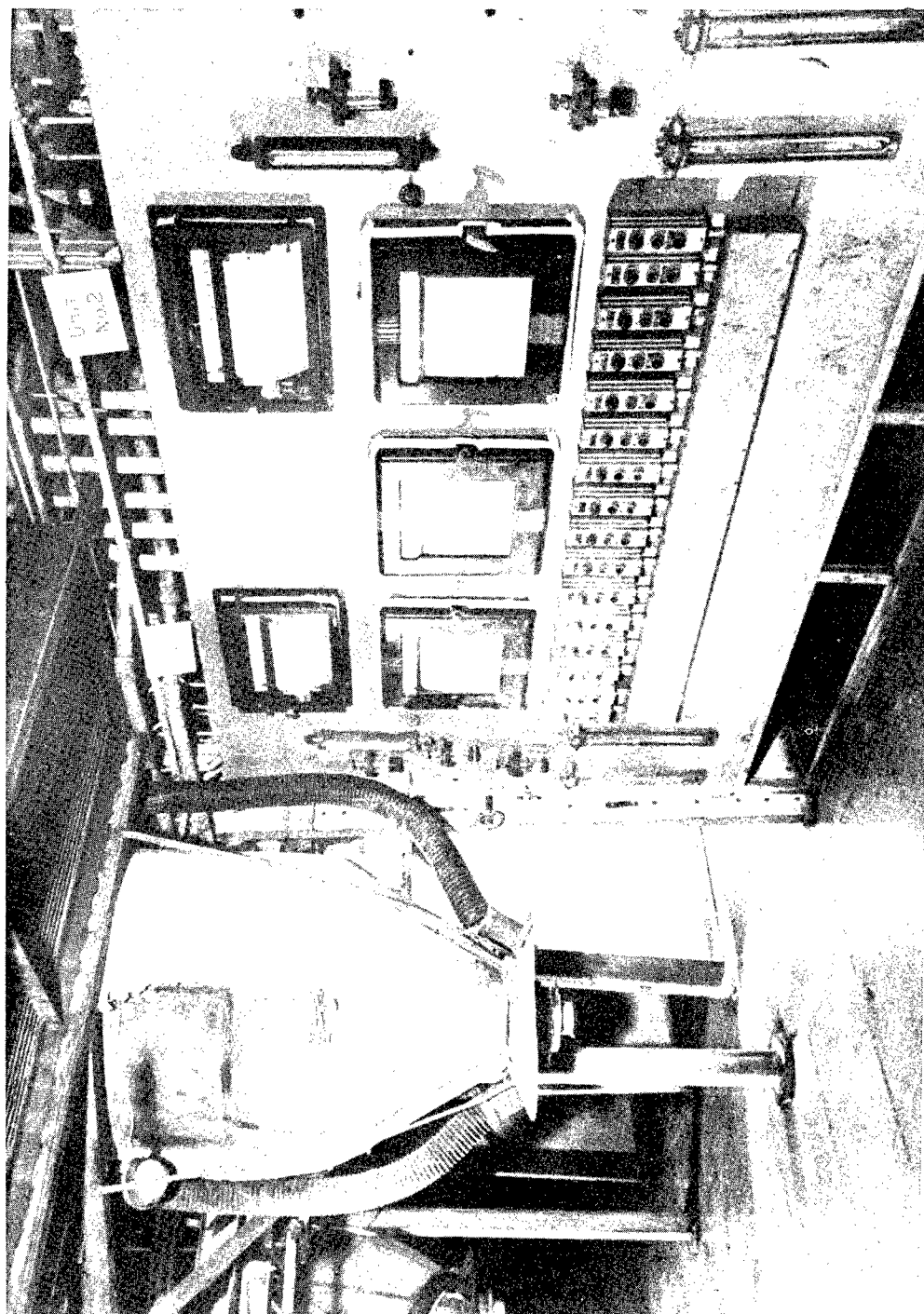


FIGURE 9. CONTROL PANEL AND CONDENSERS OF  
HORIZONTAL CHLORINATORS -FIRST FLOOR

TABLE I

## MATERIALS OF CONSTRUCTION FOR HANDLING OF PROCESS MATERIALS

<u>EXTRACTION</u>	<u>Tanks and Equipment</u>	<u>Pipe</u>	<u>Valves</u>	<u>Diaphragms</u>	<u>Pumps</u>	<u>Gaskets</u>	<u>Packing</u>	<u>Pump Lubrication</u>
Aqueous Extraction Solution, and Stripping Solution (0.2-0.5 mol HCl)	Glass-lined, Rubber-lined	Glass	Glass-lined	Tygon, Neoprene	Durchlor, Hastalloy C	Koro seal, Neoprene	Teflon, Durco 400-B	Nordcoseal 755-S Rockwell Mfg. Co., Pittsburgh
Hexone (Acid)	Glass-lined, Stoneware	Glass	Glass-lined	Neoprene	Durchlor, Hastalloy C	Neoprene	Teflon, Durco 400-B	Nordcoseal 755-S
Hexone (Neutral)*	Glass-lined, Stoneware	Glass	Glass-lined	Butyl Rubber Neoprene	-	Butyl Rubber, Neoprene	Teflon, Durco 400-B	Nordcoseal 755-S
Sulfuric Acid (5 Mol)	Glass-lined	Glass	Glass-lined	Tygon	-	Koro seal	Teflon	Nordcoseal 755-S
Sulfuric Acid (Conc.)	Black Iron	Black Iron, Glass	Black Iron	-	Black Iron, Carpenter 20 SS	-	Teflon	Nordcoseal 755-S
Conc. HCl (for Stripper Makeup)	Glass-lined	Glass	Glass-lined	Tygon	Have	Koro seal	Teflon	Nordcoseal 755-S
<u>PURIFICATION</u>								
Extraction Effluent	Glass-lined, Wood, Hastalloy C, Rubber-lined	Glass, Hard Rubber	Glass-lined, Rubber-lined	Tygon	Durchlor Hastalloy C	Koro seal	Teflon	Nordcoseal 755-S
Ammonium Phthalate Solution	SS 316	SS 316	SS 316	-	SS 316, Black Iron	Koro seal	Asbestos	Nordcoseal 755-S
Drying (to 300°C)	SS 316							Nordcoseal 755-S
Calcining (to 700°C)	Fused Quartz							Nordcoseal 755-S
<u>CHLORINATION</u>								
CCl <sub>4</sub>	SS 316	Black Iron	Black Iron		Black Iron	-	Asbestos	Nordcoseal 755-S
ZrCl <sub>4</sub> Gas (above 350° C)	Carbon, Quartz							
ZrCl <sub>4</sub> Solid (below 350° C)	Nickel							

\* Protection against acid is made since possibility of acid condition exists in most cases. Pure hexone is a good organic solvent but is not corrosive.

Background for selection is given in a report, Y-589, "Corrosion Study for a Chemical Processing Plant", Frank A. Knox, August, 1950.

In general it is found that HCNS in hexone is corrosive to about the same extent as HCl. Metals which can be used to resist this combination are Hastelloy C and Durchlor. Various rubber-like materials may be used for gasket material, although hexone is a solvent for many gasket and diaphragm materials. Butyl rubber and Neoprene appear to be the most satisfactory for resistance to neutral hexone. A large amount of process piping is standard Pyrex glass with flange fittings; this gives resistance to most of the process solutions and also provides visibility.

For resistance to sulfuric acid, glass has been used for dilute solutions. Concentrated sulfuric acid is handled in black iron, and carpenter 20 stainless steel is used as piston material in a metering pump where the piston is alternately exposed to sulfuric acid and the atmosphere.

Concentrated hydrochloric acid is handled in glass-lined tanks and glass piping. A Haveg metering pump is used for metering concentrated hydrochloric acid. Chemical resistance is good, although mechanical properties are not as satisfactory as desired.

In the phthalate purification step, an acid-resistant filter of wood is being

used. It is indicated at this time that a totally rubber-covered steel filter might be more suitable. Filter media for hydrochloric acid solutions is high temperature Vinyon or Dynell. Particle size is small and a tight weave is required.

The dryer is constructed of 316 stainless steel, which has been shown in the laboratory to be satisfactory up to 300 degrees Centigrade from the corrosion standpoint. Extensive tests on metals for calcining zirconium oxide failed to show a satisfactory metal. A fused quartz lined calciner was developed for this application in conjunction with the Bartlett-Snow Company, the Amersil Company, and the General Ceramics Company. Efficiency of this equipment will be shown by operation.

Materials of construction for zirconium chlorination are limited for zirconium tetrachloride in the gas phase. Fused quartz has been found to be resistant at very high temperatures. Carbon is good in the range of 350 to 650 degrees Centigrade. Nickel is good at 350 degrees Centigrade and below, and is fairly satisfactory up to 550 degrees Centigrade, although it gives some contamination in this range.

#### General Protection Against Corrosion

Operation of the temporary zirconium plant showed that a severe corrosion

problem can result from vapors of process solutions in the extraction and purification plants. However, general corrosion can be controlled by taking proper protective measures.

Structural supports for extraction columns are fabricated from 316 stainless steel angle and non-reusable stainless steel pipe. This stands up with only surface discoloration under the conditions present, that is, spills of dilute hydrochloric acid and vapors of HCl under oxidizing conditions.

Filters are completely enclosed and ventilated. Hoods for filters are constructed of 1/2 inch marine plywood and coated with one coat of Penkote protective coating.<sup>1</sup> Glass pipe flanges on the columns are cast iron coated with seven layers of a baked phenolic resin coating.<sup>2</sup> Nuts and bolts on flanges of columns are of stainless steel 316.

Duct work for feed makeup exhaust system is fabricated of 316 stainless steel coated with baked on Heresite. Duct work for exhaust on filter hoods is fabricated from mild steel coated with baked Heresite.

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<sup>1</sup>Penkote 500, Peninsular Chemical Product Company, Van Dyke, Mich.

<sup>2</sup>Heresite P403, Heresite Chemical Company, Manitowoc, Wis.



PROCESS CONDITIONS AND EFFICIENCY

Extraction

Present operating conditions for the extraction columns are outlined as follows:

Length of Columns (Total)

Extraction	180 Ft.
Stripping	125 Ft.
Scrubbing	65 Ft.
Thiocyanate Recovery	55 Ft.
Hexone Rate	140 GPH
CNS Concentration in Recycle Hexone	2.7 Molar
HCl Rate, Stripping Section	18-20 GPH
HCl Concentration	3.5 Molar
CNS, Concentration In	0.0 Molar
CNS, Concentration Out	2.5-3.0 Molar
Feed Rate, Zirconium Oxychloride Solution	50 GPH
HCl Concentration	1 Molar
HCNS Concentration	2.6 Molar
Zr Concentration	1 #/gal.
H <sub>2</sub> SO <sub>4</sub> Rate, Scrubber Solution	35 GPH
H <sub>2</sub> SO <sub>4</sub> Concentration	5 Normal
CNS Conc., Feed to Thiocyanate Recovery Column	1.60 Molar
CNS Conc., Discharge from Thiocyanate Recovery Column	0.1 Molar
CNS Conc., Hexone to Column	0.0 Molar
CNS Conc., Hexone from Column	2.50 Molar
Rate of Hexone to Thiocyanate Recovery Column	40 GPH
Rate of Aqueous Solution in Column	70 GPH
Conc. Hf in Raw Feed	1.5-2.0 %
Conc. Hf in Product Zr	<100 PPM
Conc. Zr in Product Hf	Approximately 2 %

Yield of Zr Product Based on Feed Solution	96%	
Percent Recycle of Hexone	96.5-97.0 %	
Percent Loss of Hexone	3-3.5 %	
Amount Makeup Hexone	90 Gals/day	
	Optimum for Extraction Section	Optimum for Stripping Section
Distribution Coefficient Hf Org/Aq	1.5	0.7
Distribution Coefficient Zr Org/Aq	0.3	0.15
Separation Factor	4-5	4-5

Operation of the extraction units is carried out to achieve the best balance between product purity and yield of zirconium. Increased purity of zirconium can be obtained at the expense of yield and Hf purity. With the present method of operation it is possible to obtain a yield of better than 96% of Zr containing less than 100 ppm Hf while obtaining hafnium product containing between 0.5% and 3.0% Zr.

#### Purification

Efficiency of the purification plant has not yet been established, and it is expected that considerable process improvement work will be required to obtain maximum efficiency. It is expected that 98 % yield of zirconium will be obtained and that product purity will be equal to, or better than, purity of product

from the initial installation based on batch operation.

Phthalate recovery is expected to be about 80 percent. Recovery efficiency is very dependent on filter operation and wash distribution on the filter.

Recycle of ammonium hydroxide from the evaporator may be a practical step for economy. It is planned to add fractionating and condensing equipment for recovery and recycle of ammonium hydroxide if it is economically justified.

#### Drying and Calcining

Operating experience with the rotary equipment is limited but serious dust losses are not anticipated. Available rotoclones and scrubbers will be activated if necessary.

#### Operating Costs

Typical operating costs are given in the following tables. Table II gives the cost for  $ZrO_2$  production in the month of January, 1951. Table III gives cost for  $ZrO_2$  production total for the fiscal year July, 1950 through April, 1951.

These costs resulted from operation of the temporary zirconium production

facilities. Considerable economies in both labor and materials are expected from operation of the permanent zirconium plant. Estimated costs in report Y-573, p10, are expected to be in line with actual cost if allowance is made for general price advances.

TABLE IIUNIT COST OF ZRO<sub>2</sub> PRODUCTION, JANUARY, 1951

	Total Cost	Cost Per Pound Zr Produced
	<u>\$93,523</u>	<u>\$3.002</u>
<b>Material</b>		
Ammonium Hydroxide	1584	.051
Lime	133	.004
Hydrochloric Acid	1874	.061
Salicylic Acid	38934	1.251
Sulfuric Acid	658	.021
Ammonium Thiocyanate	10057	.323
ZrCl <sub>4</sub>	35165	1.129
Hexone	2186	.070
Natural Gas	693	.022
Steam	1534	.049
Treated Water	536	.017
Electricity	134	.004
Operating Labor, Direct	13,667	.439
Maintenance, Labor, & Material	15,763	.506
Allocated Plant Expense	12,455	.400
Analytical	3,740	.120
Miscellaneous *	9,128	.293
Total	<u>\$148,276</u>	<u>\$4.761</u>

Pounds Zirconium as Oxide Produced - 31,134

\* Protective Clothing, Shipping Charges, Janitorial Services, Etc.

TABLE III

UNIT COST ZrO<sub>2</sub> PRODUCTION FROM JULY, 1950 THRU  
APRIL, 1951

Material	<u>Total Cost</u>	<u>Cost/lb.</u>
	<u>\$731,971</u>	<u>\$2.943</u>
Ammonium Hydroxide	\$13,041	.052
Lime	1,710	.007
Hydrochloric Acid	22,408	.090
Salicylic Acid	278,764	1.121
Sulfuric Acid	3,679	.015
Ammonium Thiocyanate	89,211	.359
ZrCl <sub>4</sub>	287,050	1.154
Hexone	14,985	.060
Caustic Flake	41.	.000
Natural Gas	5,807	.023
Steam	10,821	.044
Treated Water	3,280	.013
Electricity	1,174	.005
Operating Labor	110,385	.443
Maintenance, Labor, & Material	148,453	.596
Allocated Plant Expense	124,067	.498
Analytical	33,471	.135
Miscellaneous	<u>88,920</u>	<u>.357</u>
Total	<u>\$1,237,267</u>	<u>\$4.972</u>

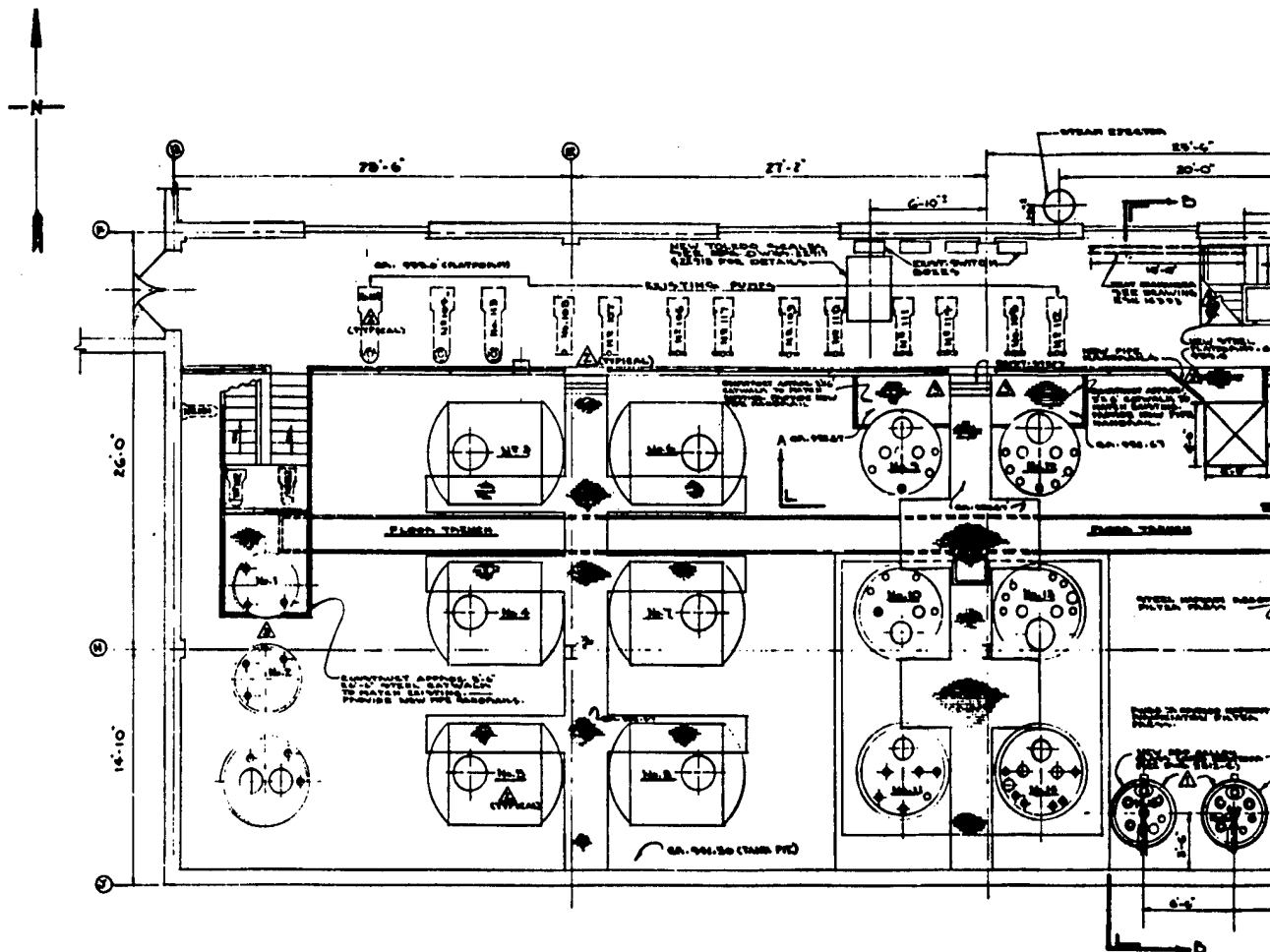
Pounds Zirconium as Oxide Produced - 248,751

### BIBLIOGRAPHY OF Y-12 LITERATURE BEARING ON PRODUCTION OF ZIRCONIUM MATERIALS

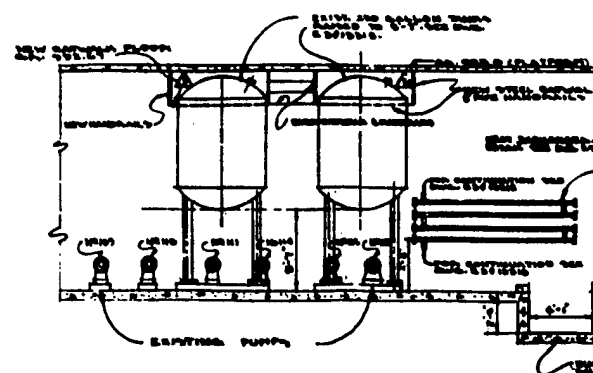
Bibliographies of Y-12 reports and reports of the MIT Practice School (Y-B4-43 and Y-B4-44) have been prepared by Mrs. Frances Sachs of the Y-12 Technical Information Center. Reports listed in these bibliographies contain important background material regarding the present processes for extraction, purification, and chlorination of zirconium materials at Y-12.

### CONSTRUCTION DRAWINGS

Reduced drawings are given of principal engineering designs used in construction of the permanent zirconium plant. Drawings were prepared by Mr. F. S. Patton of the Engineering Department at Y-12 and were used as the basis of field instruction to construction personnel.



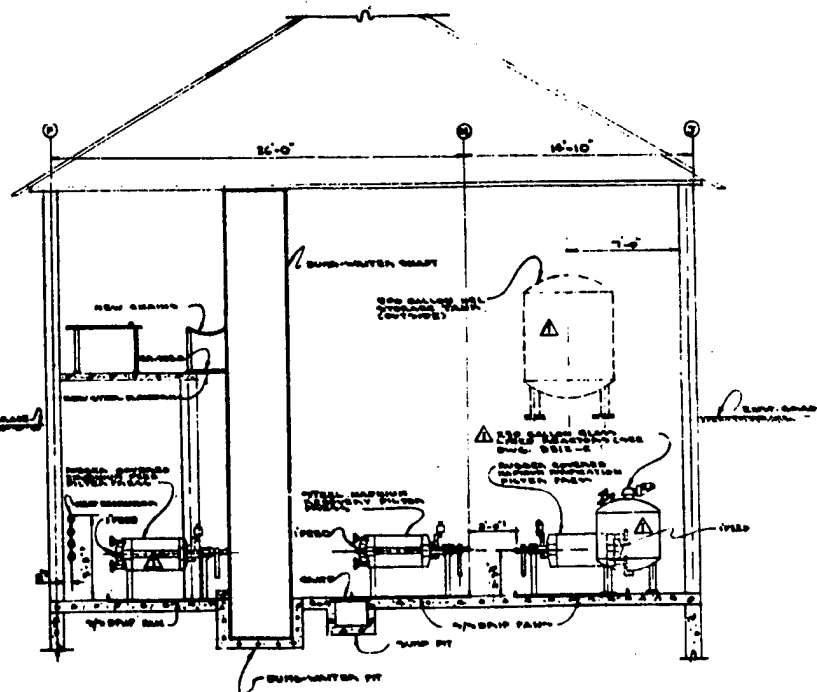
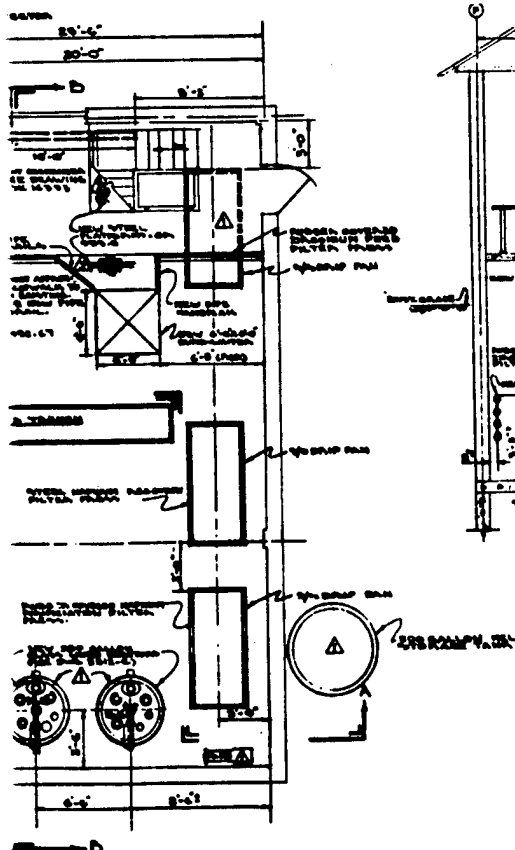
TANK FARM PLAN



ELEVATION-AA


PERMANENT ZIRCONIUM PLANT EQUIPMENT LOCA





ELEVATION—BB

## TANK SCHEDULE

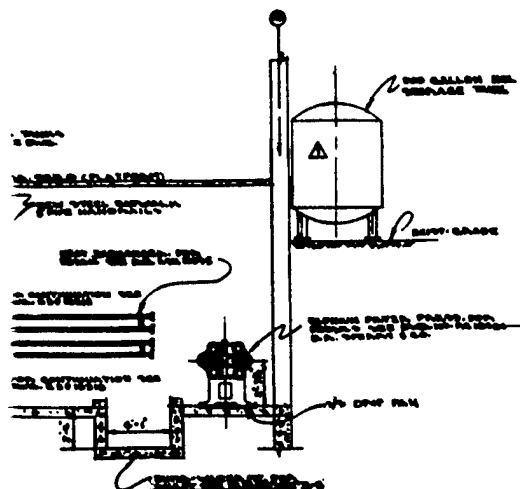
- 
 1,6,7,8 ————— PHTHALATE STORAGE  
 2 ————— NH<sub>4</sub>OH  
 3 ————— FEED STORAGE  
 4,5,6 ————— PRODUCT STORAGE  
 9,10,11,12 — FEED HANDLING AREA  
 11,12,13,14 — MAGNUM

REF. DW'GS.

- E 131145 — PERMANENT ZIRCONIUM PLANT  
 FEED MAKEUP AREA — FLOW DIAGRAM.  
 E 1011346 — PERMANENT ZIRCONIUM PLANT.  
 FEED MAKEUP AREA — WHITE COVERED.  
 E 2011349 — PERMANENT ZIRCONIUM PLANT.  
 FEED MAKEUP AREA — HEAT EXCHANGER.  
 E 2011348 — PERMANENT ZIRCONIUM PLANT.  
 FEED MAKEUP AREA — FROD SUMMIT AFTER.  
 E 2011345 — PERMANENT ZIRCONIUM PLANT.  
 FEED MAKEUP AREA — REACTOR SUMMIT AFTER.  
 C 2011347 — PERMANENT ZIRCONIUM PLANT.  
 FEED MAKEUP AREA — SCALE'S EXHAUST HOOD.  
 E 131024 — TYPE S.C. FILTER PRESS, D.G.  
 TERRY & CO.  
 2312 — 100 GALLON REACTOR — GLASS  
 COLE PRODUCTS, INC.  
 2217 — PIT LAYOUT — TYPE 9900 — TOLSOO  
 SCALE.  
 2218 — FRAME DETAILS — TYPE 9900  
 TOLSOO SCALE.

## PJMP SCHEDULE

- [illegible]



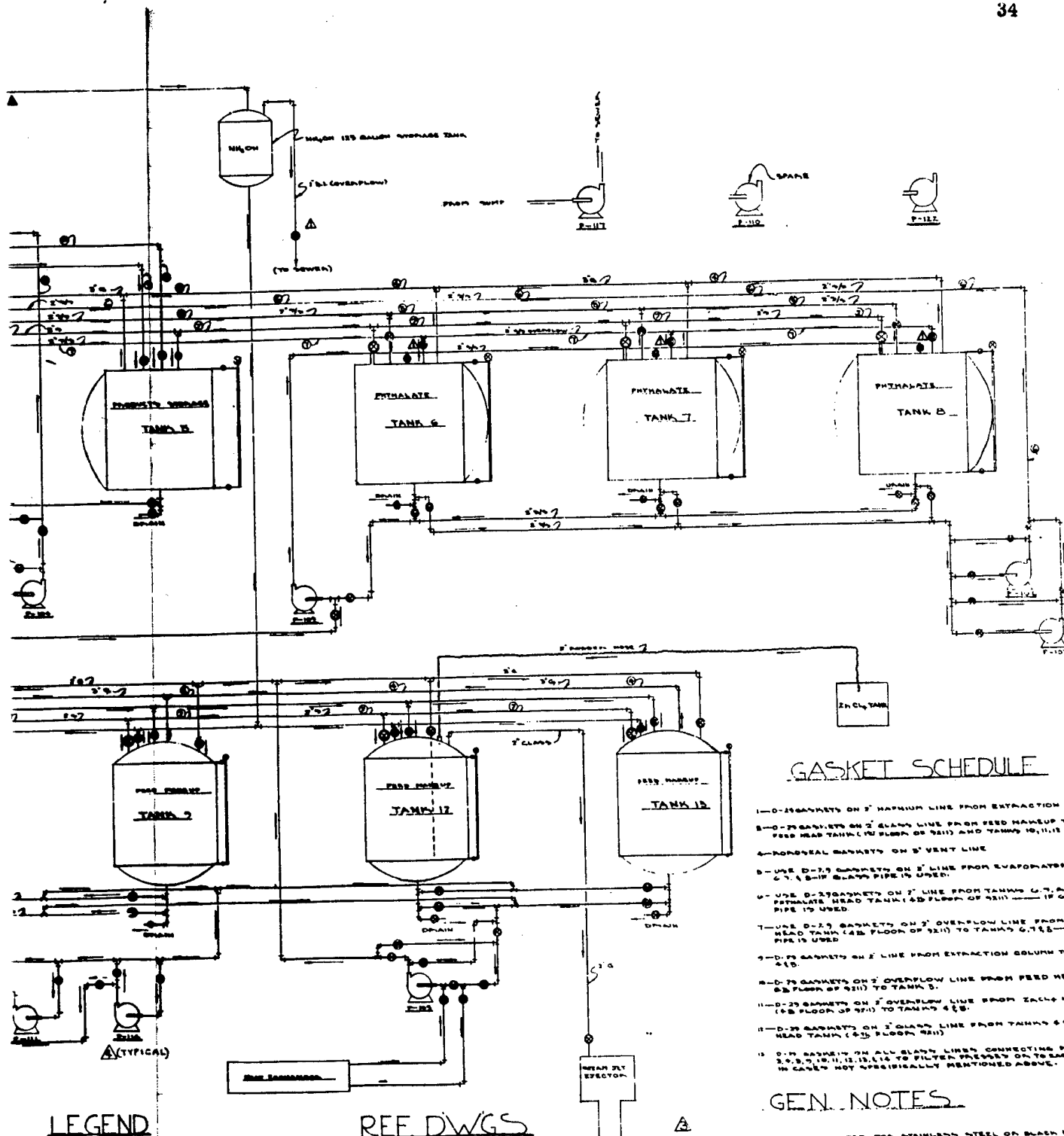
GEN. NOTES.

1. DIMENSIONS SHOWN MAY BE VARIED TO MEET FIELD CONDITIONS.
2. LIFTING EQUIPMENT SHOWN IN LIGHT LINE WHILE NEW EQUIPMENT IN HEAVY.
3. FILTERS TO BE EQUIPPED WITH SUITABLE FEED DISK PAIR CONTACTED AT F.O.S.
4. FOR FRAME & PIT DETAILS OF TOLDO SEAL-SEE MANUFACTURER'S DRAWINGS 22175.
5. FOR LOCATION OF SCALE-SEE DRAWING 24K 14667.
6. DUMP WATER REMOVED FROM SLIDE 9201 IS INSTALLED IN BUILDING 9211 FOR GENERAL ILLUSTRATIONS SEE BOTH IN WS 249955.
7. FOR EXHAUST TO FEED MAKEUP AREA-SEE DWG. 24K 14647, EN 14653 & CEN.

IPMENT LOCATION-FEED MAKEUP AREA, PLAN & ELEVATIONS

2





## GASKET SCHEDULE

- 1-0-25 GASKETS ON 2" NITROGEN LINE FROM EXTRACTION COLUMN.
- 2-0-25 GASKETS ON 2" GLASS LINE FROM FEED MAKEUP TANKS TO FEED HEAD TANK (10' FLOOR OF 9211) AND TANKS 10, 11, 12, 13.
- 3-0-25 GASKETS ON 2" VENT LINE
- 4-USE 0-25 GASKETS ON 2" LINE FROM EVAPORATOR TO TANKS 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13.
- 5-USE 0-25 GASKETS ON 2" LINE FROM TANKS 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13 TO FEED HEAD TANK (10' FLOOR OF 9211) IF GLASS PIPE IS USED.
- 6-USE 0-25 GASKETS ON 2" OVERFLOW LINE FROM PHOSPHATE HEAD TANK (10' FLOOR OF 9211) TO TANKS 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13 IF GLASS PIPE IS USED.
- 7-USE 0-25 GASKETS ON 2" OVERFLOW LINE FROM PHOSPHATE HEAD TANK (10' FLOOR OF 9211) TO TANKS 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13 IF GLASS PIPE IS USED.
- 8-0-25 GASKETS ON 2" LINE FROM EXTRACTION COLUMN TO TANKS 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13.
- 9-0-25 GASKETS ON 2" OVERFLOW LINE FROM FEED HEAD TANK (10' FLOOR OF 9211) TO TANKS 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13.
- 10-0-25 GASKETS ON 2" OVERFLOW LINE FROM TANKS 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13 TO FEED HEAD TANK (10' FLOOR OF 9211).
- 11-0-25 GASKETS ON 2" GLASS LINE FROM TANKS 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13 TO FEED HEAD TANK (10' FLOOR OF 9211).
- 12-0-25 GASKETS ON ALL GLASS LINES CONNECTING TANKS 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13 TO FEED HEAD TANK (10' FLOOR OF 9211) OR TO EACH OTHER IN CASES NOT SPECIFICALLY MENTIONED ABOVE.

## GEN. NOTES

- 1-GLASS PIPE MAY BE SUBSTITUTED FOR STAINLESS STEEL OR BLACK IRON PIPE WHENEVER IT IS MORE EXPEDIENT.
- 2-ALL PIPING SHOWN DIAGNOSTICALLY ONLY. EXACT LOCATION AND METHOD OF SUPPORT TO BE DETERMINED IN THE FIELD.
- 3-FITTED ALL PIPE TO LOW POINTS FOR DRAINING LINES AND TO AVOID AIR LOCKS IN PUMP SUCTION AND DISCHARGE LINES.
- 4-ALL VALVES ON GLASS LINES TO BE HILLS-HESANNA VALVES. GLASS LINES.
- 5-ALL PIPING TO BE 2" FREE GLASS EXCEPT WHERE OTHERWISE NOTED.
- 6-USE HILLS-HESANNA STAINLESS STEEL DIAPHRAGM VALVES ON STAINLESS STEEL LINES. BRASS OR STEEL GATE VALVES ON BLACK IRON LINES.
- 7-SEE LOCATION OF EQUIPMENT IN THE FEED MAKEUP AREA. SEE DWG. E 101 13213 FOR CONTINUATION OF LINES.
- 8-SEE DWG. E 101 13213, E 101 13214, E 101 13215, E 101 13216, E 101 13217, E 101 13218, E 101 13219, E 101 13220, E 101 13221, E 101 13222, E 101 13223.

## LEGEND

- INDICATES STAINLESS STEEL PIPE
- INDICATES GLASS PIPE
- INDICATES FEED GLASS PIPE
- INDICATES BLACK IRON PIPE
- INDICATES ALL TYPES OF VALVES
- INDICATES RUBBER HOSE
- INDICATES RUBBER HOSE (FOR VESSELS)
- INDICATES DIRECTION OF FLOW
- INDICATES PUMP NUMBER/RELATIVE PUMP SCHEDULE

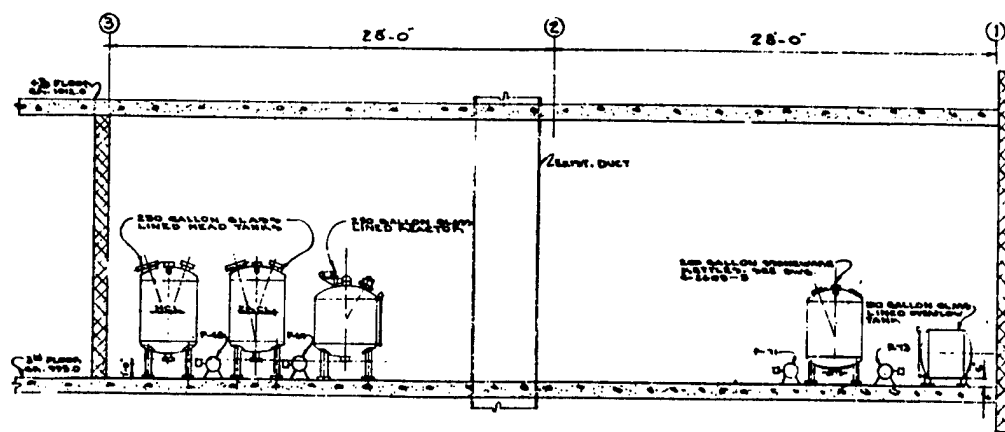
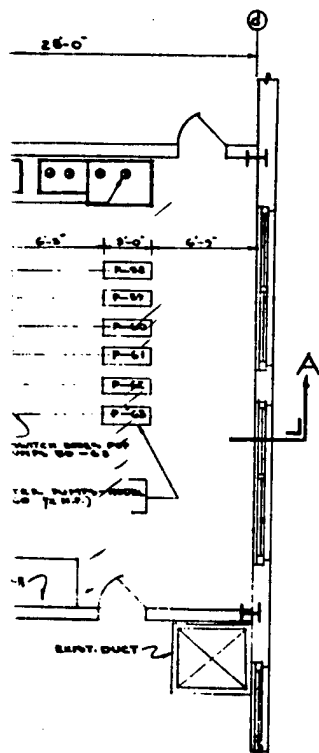
## REF DWGS

- E 101 13213 — PERMANENT ZIRCONIUM PLANT EQUIPMENT LOCATION — FEED MAKEUP AREA
- E 101 13214 — PERMANENT ZIRCONIUM PLANT — PIPING LAYOUT — PURIFICATION AREA
- E 101 13215 — PERMANENT ZIRCONIUM PLANT — PIPING LAYOUT — EXTRACTION CONTROL AREA
- E 101 13216 — PERMANENT ZIRCONIUM PLANT — GENERAL PIPING LAYOUT
- E 101 13217 — PERMANENT ZIRCONIUM PLANT — FEED MAKEUP AREA
- E 101 13218 — PERMANENT ZIRCONIUM PLANT — EXTRACTION COLUMN

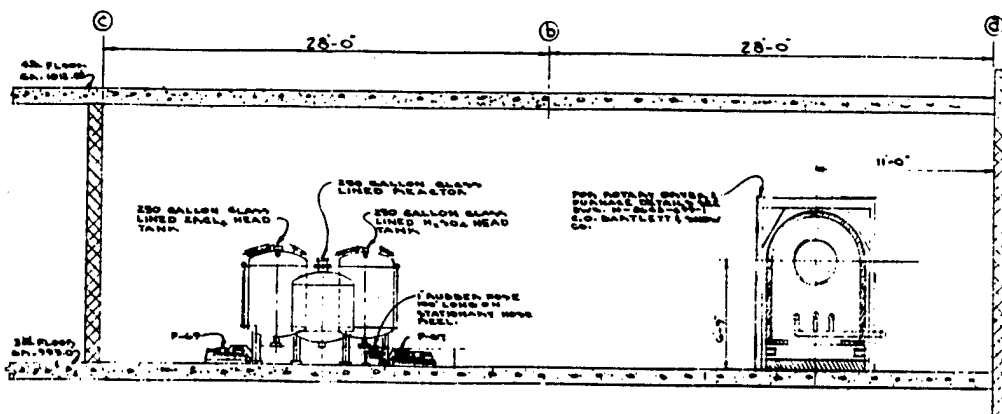
PERMANENT ZIRCONIUM PLANT FLOW DIAGRAM, FEED MAKEUP AREA

②





SECTION-BB



SECTION-CC

PUMP SCHEDULE

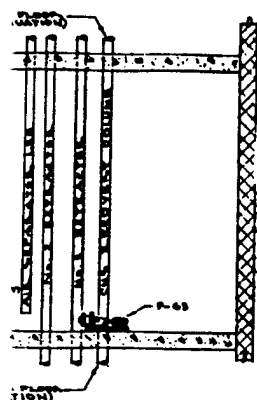
- P-50 - P-53 DUNHLOP 2-1/2" x 60 (1/2")  
 P-54 MILTON ROY DUPLEX  
 P-55 MILTON ROY DUPLEX  
 P-56 MILTON ROY DUPLEX  
 P-57 MILTON ROY DUPLEX  
 P-58 MILTON ROY DUPLEX  
 P-59 MILTON ROY DUPLEX  
 P-60 MILTON ROY DUPLEX  
 P-61 MILTON ROY DUPLEX  
 P-62 MILTON ROY DUPLEX  
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 P-96 MILTON ROY DUPLEX  
 P-97 MILTON ROY DUPLEX  
 P-98 MILTON ROY DUPLEX  
 P-99 MILTON ROY DUPLEX  
 P-100 MILTON ROY DUPLEX

REF. DWG'S

- E101355 - PERMANENT ZIRCONIUM PLANT -  
 EQUIPMENT LOCATION - PURIFICATION &  
 CALCING AREA - PLAN & SECTIONS  
 E101356 - PERMANENT ZIRCONIUM PLANT -  
 FLOW DIAGRAM - EXTRACTION COLUMN  
 E101357 - PERMANENT ZIRCONIUM PLANT -  
 PIPING LAYOUT - EXTRACTION COLUMN  
 E101358 - PERMANENT ZIRCONIUM PLANT -  
 EQUIPMENT BASES - 2nd FLOOR  
 D101359 - PERMANENT ZIRCONIUM PLANT -  
 FILTER PRESS EXHAUST SYSTEM  
 D101360 - PERMANENT ZIRCONIUM PLANT -  
 ONE LINE DIAGRAM - EXTRACTION  
 COLUMN AREA  
 H-5643-672-1 - ROTARY DRYER - S.O.  
 BARTLETT & SNOOK CO.  
 H-5643-672-2 - PERLEY FILTER PRESS -  
 D.R. PERLEY & CO.  
 C-5605-7 - 250 GALLON WITHE WARE  
 KETTLE - GENERAL CERAMIC CORP.  
 5512-C - 250 GALLON GLASS LINED  
 REACTOR - GLASSLINE PRODUCTS  
 G7555-1 - 250 GALLON EFFLUENT HEAD  
 TANK - ALLOY FABRICATORS, INC.

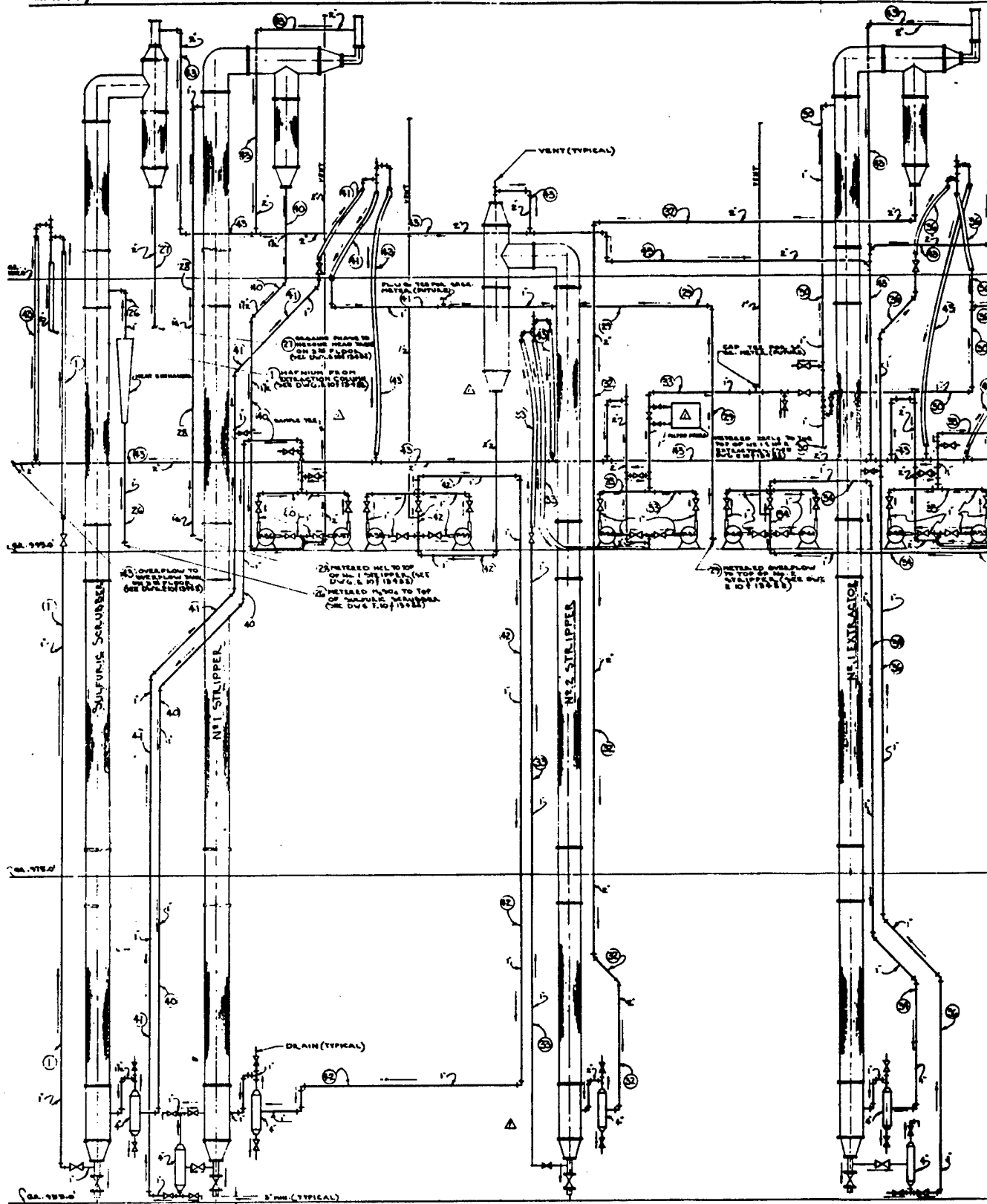
GEN. NOTES

- 1 - ALL EQUIPMENT SHOWN ON 2nd FLOOR TO  
 BE NEW EXCEPT AS NOTED.  
 2 - ALL TANK SUPPORTS TO BE DESIGNED AND  
 INSTALLED BY THE FIELD.  
 3 - DIMENSIONS SHOWN ARE APPROXIMATE  
 AND MAY BE VARIED TO MEET REQUIREMENTS.  
 4 - SEE MANUFACTURER'S DRAWINGS FOR DETAILS  
 OF ROTARY DRYER, FILTER PRESS, 1000 GAL.  
 WITHE WARE KETTLE, GLASS LINED REACTOR,  
 250 GALLON HEAD TANK.  
 5 - NEW DUCT, 10" DIA. STEEL, 10' LONG, 10' DIA.  
 BUT EXACT LOCATION TO BE DETERMINED  
 BY FIELD.  
 6 - ALL FILTERS TO BE EQUIPPED WITH EITHER  
 STAINLESS STEEL OR HASTELLOY C-276  
 FRAME AND 10" DIA. 10' LONG, 10' DIA.  
 OF DRYER FRAME TO BE DETERMINED BY FIELD.  
 7 - TITRATION TABLE TO BE DESIGNED (CONSTRUCTED  
 BY FIELD).  
 8 - WATER BODIES FOR PUMPS 50-75 TO  
 BE LOCATED OUTSIDE BUILDING AND  
 MAY BE USED TO MEET FIELD REQUIREMENTS.  
 9 - PERMANENT PIPING (FUTURE) TO BE FOR  
 SPECIFIC GRAVITY METERS.  
 10 - ALL TANKS TO HAVE NIGHT CLIMBERS  
 AND OR NIGHT CLIMBERS, DEPENDS  
 UPON AVAILABLE OPENINGS.  
 11 - PER FILTER PRESS EXHAUST SYSTEM  
 SET OUT ONE HOLE.



PERMANENT ZIRCONIUM PLANT EXTRACTION CONTROL AREA EQUIPMENT LOCATION-PLAN &amp; SECTION

②



FLOW DIAGRAM

# PIPE MARK SCHEDULE

- ① RECOVERED H<sub>2</sub>O<sub>2</sub> THRU P-66 TO TOP OF THE SULFURIC SCRUBBER.
- ② ORGANIC PHASE FROM TOP OF THE SULFURIC SCRUBBER TO WASTE HEAD TANK.
- ③ RECOVERED HCL THRU P-68 TO TOP OF THE H<sub>2</sub> STRIPPER.
- ④ RECOVERED OVERFLOW THRU P-75 TO TOP OF THE H<sub>2</sub> STRIPPER.
- ⑤ RECOVERED ZELCO THRU P-69 TO TOP OF H<sub>2</sub> STRIPPER.
- ⑥ RECOVERED WASTE THRU P-71 TO BOTTOM OF THE CH<sub>3</sub> RECOVERY COLUMN (THRU H<sub>2</sub> STRIPPER).
- ⑦ ORGANIC PHASE FROM TOP OF H<sub>2</sub> STRIPPER TO BOTTOM OF H<sub>2</sub> STRIPPER.
- ⑧ AQUEOUS PHASE FROM BOTTOM OF THE H<sub>2</sub> STRIPPER (THRU GRAVITY LEG & PUMP 80-81) TO TOP OF H<sub>2</sub> STRIPPER.
- ⑨ ORGANIC PHASE FROM AIR SEPARATOR LEG OF H<sub>2</sub> STRIPPER THRU PUMP 80-81 TO BOTTOM OF H<sub>2</sub> STRIPPER.
- ⑩ AQUEOUS PHASE FROM BOTTOM OF H<sub>2</sub> STRIPPER (THRU GRAVITY LEG) TO TOP OF THE H<sub>2</sub> STRIPPER.
- ⑪ AQUEOUS PHASE FROM BOTTOM OF H<sub>2</sub> STRIPPER TO BOTTOM OF H<sub>2</sub> STRIPPER.
- ⑫ AQUEOUS PHASE FROM BOTTOM OF H<sub>2</sub> STRIPPER (THRU GRAVITY LEG) TO TOP OF THE CH<sub>3</sub> RECOVERY COLUMN.
- ⑬ ORGANIC PHASE FROM AIR SEPARATOR LEG OF CH<sub>3</sub> RECOVERY COLUMN (THRU PUMP 84-85) TO BOTTOM OF H<sub>2</sub> STRIPPER.
- ⑭ ORGANIC PHASE FROM TOP OF H<sub>2</sub> STRIPPER (THRU PUMP 80-81) TO BOTTOM OF H<sub>2</sub> STRIPPER.
- ⑮ AQUEOUS PHASE FROM BOTTOM OF H<sub>2</sub> STRIPPER (THRU GRAVITY LEG) TO TOP OF THE H<sub>2</sub> STRIPPER.
- ⑯ ORGANIC PHASE FROM AIR SEPARATOR LEG OF H<sub>2</sub> STRIPPER (THRU PUMP 80-81) TO BOTTOM OF H<sub>2</sub> STRIPPER.
- ⑰ OVERFLOW FROM ALL EXTRACTION COLUMNS AND GRAVITY LEGS TO THE OVERFLOW TANK ON 2<sup>ND</sup> FLOOR.
- ⑱ ZELCO FROM BOTTOM OF THE CH<sub>3</sub> RECOVERY COLUMN (THRU GRAVITY LEG & PUMP 84-85) TO FILL MAKE UP AREA IN THE TANK ROOM ON THE PURIFICATION AREA ON THE 2<sup>ND</sup> FLOOR.
- ⑲ WASTE FROM BOTTOM OF THE SULFURIC SCRUBBER (THRU GRAVITY LEG) TO FILL MAKE UP AREA IN THE TANK ROOM.

## GENERAL NOTES

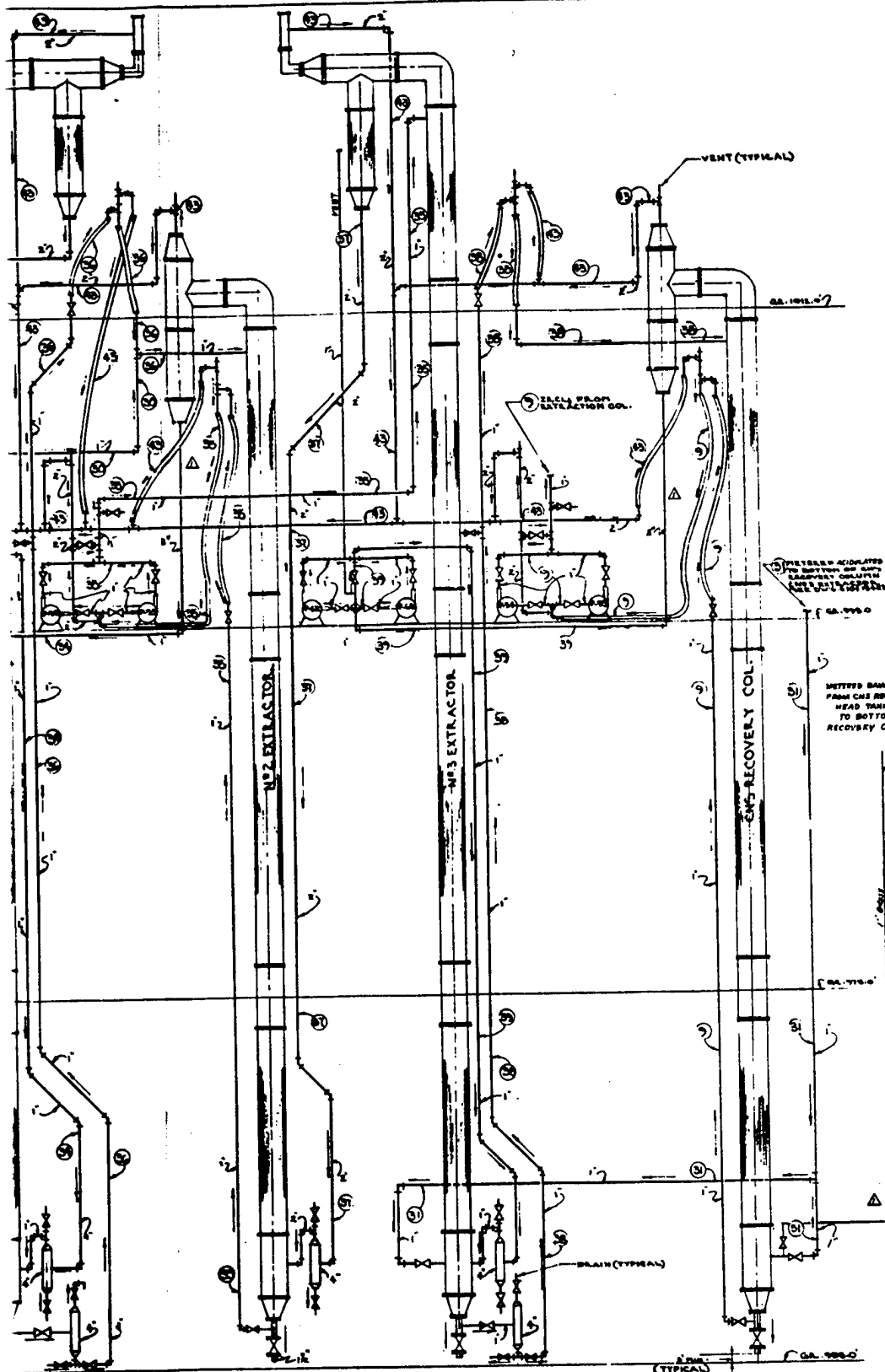
- 1-ALL PIPING SHOWN DIAGRAMMATICALLY ONLY.
- 2-ALL PIPING TO BE PYREX GLASS LINED.
- 3-ALL VALVES TO BE MILLS-MECANNA GLASS LINED VALVES.
- 4-ALL MILLS-MECANNA GLASS LINED VALVES ON THE 1<sup>ST</sup> FLOOR TO BE NICKEL-PLATED.
- 5-USE D-25 GASKETS ON ALL GLASS LINES.
- 6-USE 247 WELDING ROD WHEN WELDING TO 1" IN THE CONNECTION OF COLUMN SUPPORTS.
- 7-ELEVATION OF OVERFLOW LINE ON 2<sup>ND</sup> FLOOR IS 1000.0 (ATCH OVER FLOOR LINE TO WARD OVERFLOW TANK (SEE DWG 1001280).
- 8-PUMP 80-81 TO BE DUAL-CHLOR. 2-210-60 (1-H.P.) BOOSTER PUMP FOR LOCATION OF PUMP SEE DWG. E 1011545.
- 9-FOR CONTINUATION OF LINES (1), 2, 26, 27, 28, 29, 30 & 31 - SEE DWG. E 1011545, E 1011546, E 1011547 & E 1011548.
- 10-ALL COLUMNS TO BE 4" PYREX GLASS PIPE EXCEPT FOR ONE (1) 12" SECTION OF 6" GLASS PIPE & 12" SPECIAL REDUCING ALL AT THE TOP OF ALL COLUMNS.

## REF. D.W.G.

- E 1011545 - PERMANENT ZIRCONIUM PLANT - FLOW DIAGRAM - FILL MAKEUP AREA.
- E 1011546 - PERMANENT ZIRCONIUM PLANT - PIPING LAYOUT - PURIFICATION AREA.
- E 1011547 - PERMANENT ZIRCONIUM PLANT - PIPING LAYOUT - EXTRACTION CONTROL AREA.
- E 1011548 - PERMANENT ZIRCONIUM PLANT - GENERAL PIPING LAYOUT.

## LEGEND

- (1), (2), (3), (4), (5) PIPE MARK SCHEDULE.
- (6) DUAL-CHLOR. 2-210-60 (1-H.P.) BOOSTER PUMP.
- (7) RUDDER GRAVITY LEG.

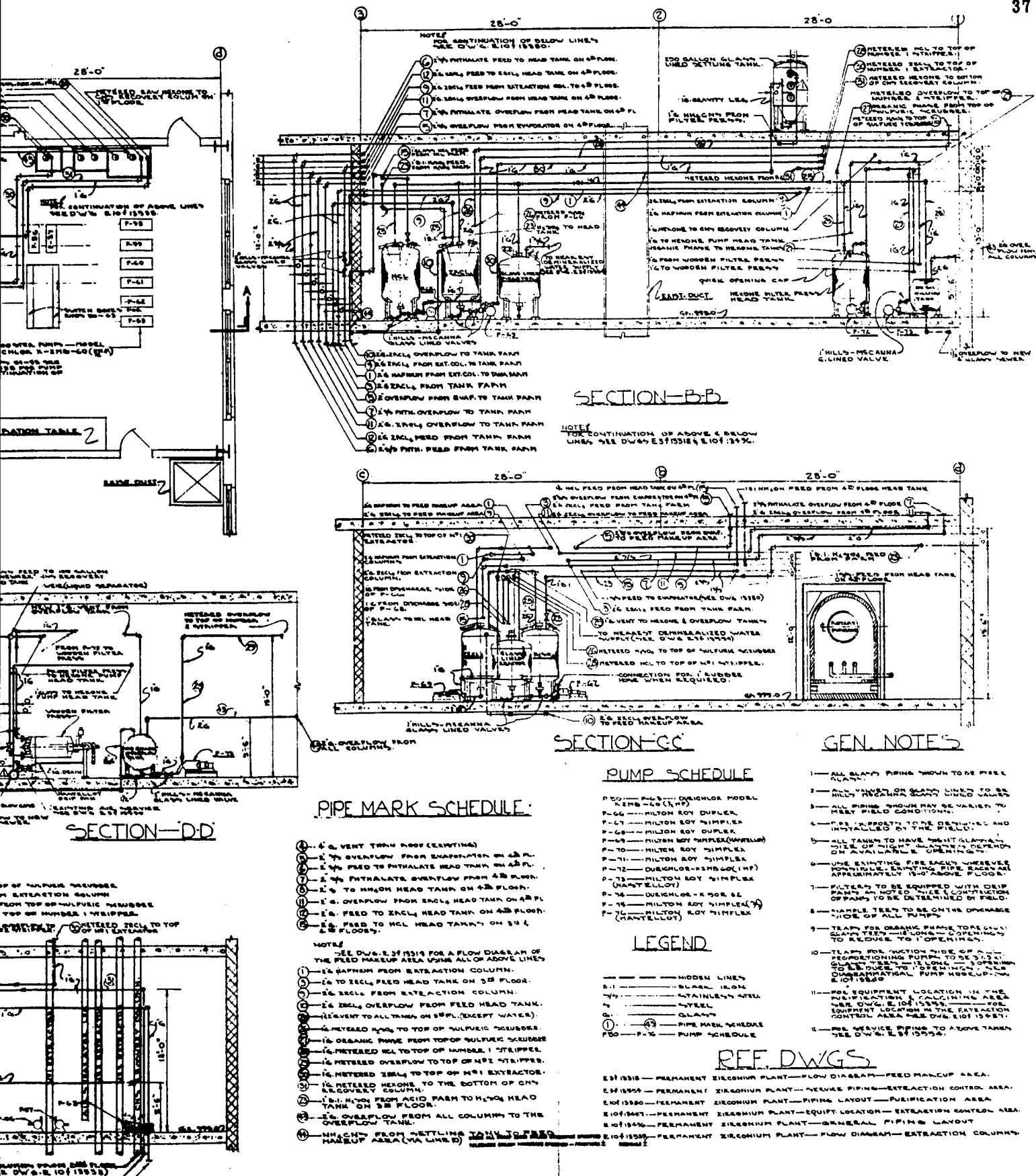


PERMANENT ZIRCONIUM PLANT FLOW DIAGRAM EXTRACTION COLUMNS

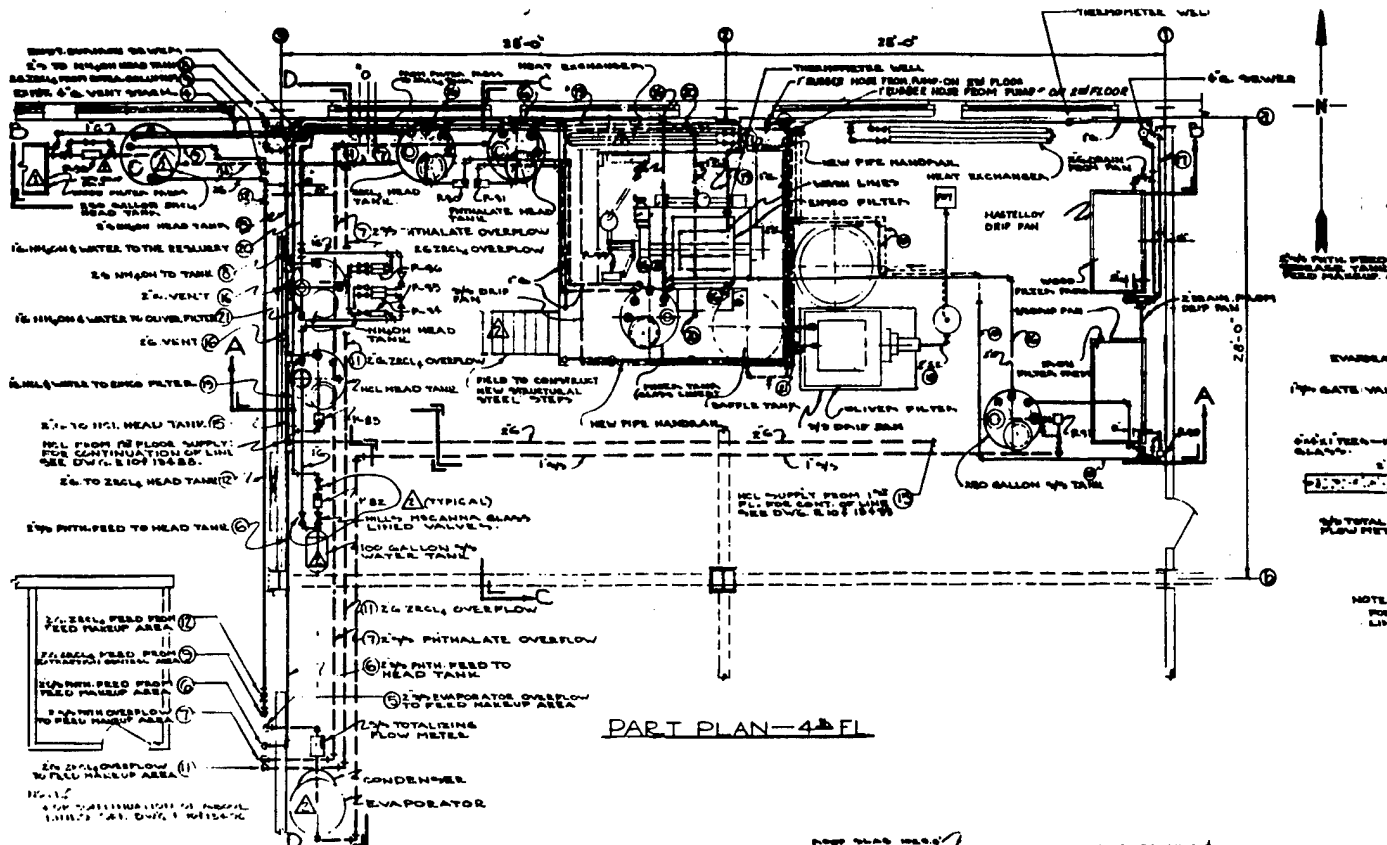
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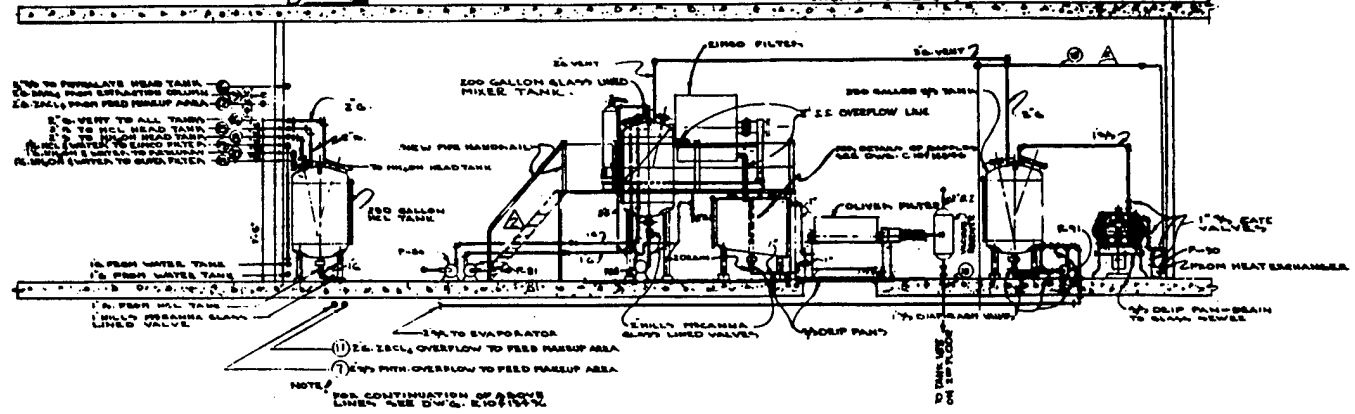




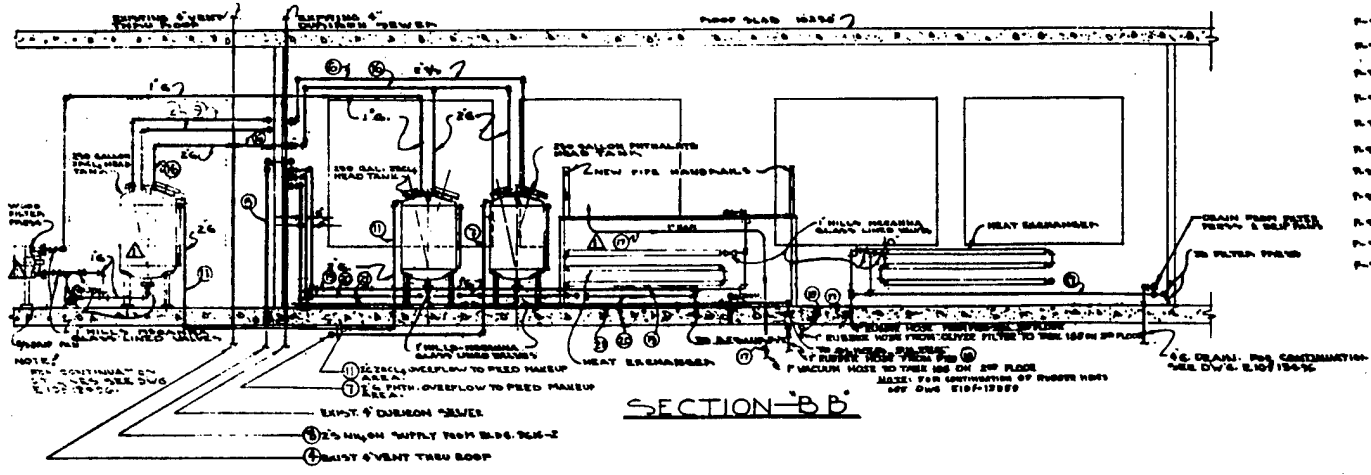
PERMANENT ZIRCONIUM PLANT PIPING LAYOUT-EXTRACTION CONTROL AREA, PLANS &amp; SECTIONS



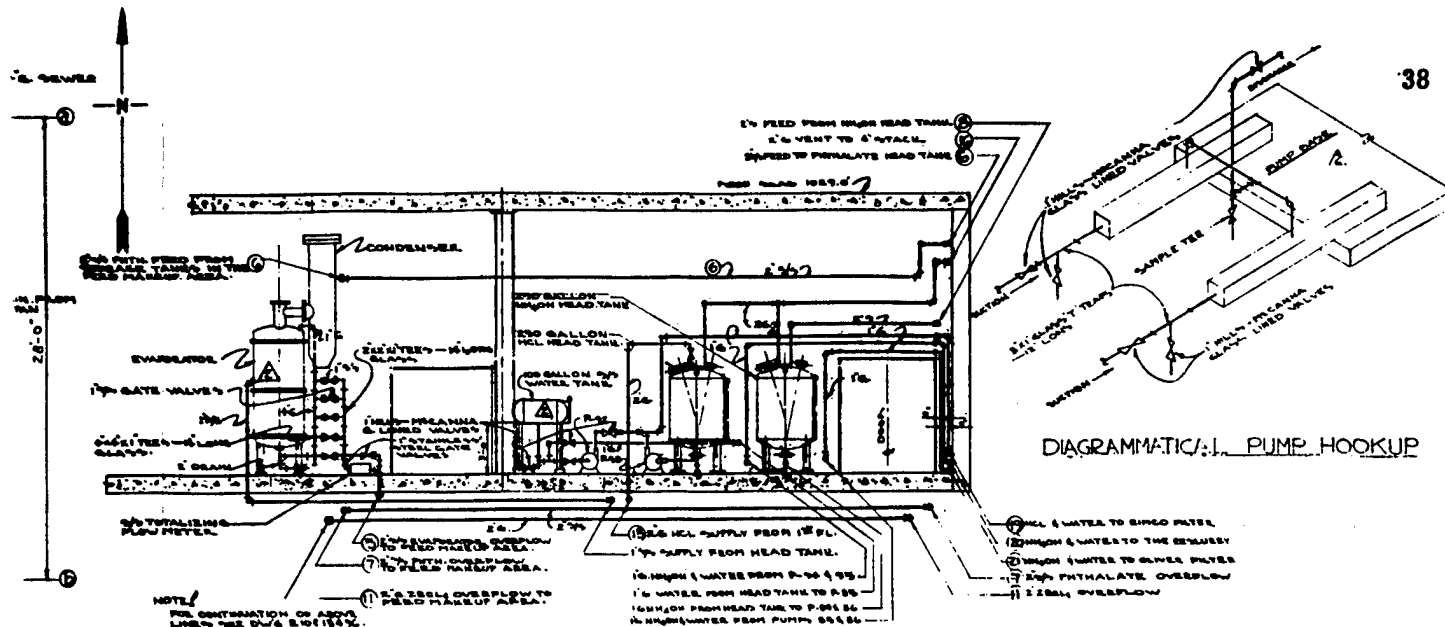
PART PLAN-4 FL



SECTION-AA



SECTION-BB



SECTION-DD

## LEGEND

- 3/8" — STAINLESS STEEL
- 2" — STEEL
- 2" — PYREX GLASS PIPE
- 2" — BLACK IRON
- FLEXIBLE RUBBER HOSE
- P-30, P-32 PUMP NUMBERS (SEE PUMP SCHEDULE)
- ④ — ② PIPE NUMBERS (SEE PIPE SCHEDULE)
- MIDDLE LINES (UNDER FLOOR OR PLATFORM)

## REF. DWG'S

- E-101555 — PERMANENT ZIRCONIUM PLANT — EQUIPMENT LOCATION
- E-101556 — PERMANENT ZIRCONIUM PLANT — PURIFICATION AREA
- E-101557 — PERMANENT ZIRCONIUM PLANT — GENERAL PIPING LAYOUT
- E-101558 — PERMANENT ZIRCONIUM PLANT — PIPING LAYOUT — EXTRACTION CONTROL AREA

## SECTION-CC

## PIPE MAP SCHEDULE

- ① 2" INCH FROM EXTRACTION COLUMN
- ② 2" INCH FROM 1" INCH TO EXHAUST 2" INCH
- ③ 2" INCH FROM EVAPORATOR ON 2ND FLOOR
- ④ 2" INCH FEED TO PHthalate HEAD TANK ON 2ND FLOOR
- ⑤ 2" INCH PHthalate OVERFLOW FROM 2ND FLOOR
- ⑥ 2" INCH FEED TO NH<sub>4</sub>OH HEAD TANK ON 2ND FLOOR
- ⑦ 2" INCH OVERFLOW FROM 2ND FLOOR
- ⑧ 2" INCH FEED TO 2ND FLOOR
- ⑨ 2" INCH FEED TO 2ND FLOOR
- ⑩ 2" INCH FEED TO 2ND FLOOR
- ⑪ 2" INCH FEED TO 2ND FLOOR
- ⑫ 2" INCH FEED TO 2ND FLOOR
- ⑬ 2" INCH FEED TO 2ND FLOOR
- ⑭ 2" INCH FEED TO 2ND FLOOR
- ⑮ 2" INCH FEED TO 2ND FLOOR
- ⑯ 2" INCH FEED TO 2ND FLOOR
- ⑰ 2" INCH FEED TO 2ND FLOOR
- ⑱ 2" INCH FEED TO 2ND FLOOR
- ⑲ 2" INCH FEED TO 2ND FLOOR
- ⑳ 2" INCH FEED TO 2ND FLOOR

## NOTE

- SEE DWG. E-101555 FOR A FLOW DIAGRAM OF THE PIPING MAP AREA USING ALL OF THE ABOVE LINES
- ① 2" INCH FEED TO 2ND FLOOR
- ② 2" INCH FEED TO 2ND FLOOR
- ③ 2" INCH FEED TO 2ND FLOOR
- ④ 2" INCH FEED TO 2ND FLOOR
- ⑤ 2" INCH FEED TO 2ND FLOOR
- ⑥ 2" INCH FEED TO 2ND FLOOR
- ⑦ 2" INCH FEED TO 2ND FLOOR
- ⑧ 2" INCH FEED TO 2ND FLOOR
- ⑨ 2" INCH FEED TO 2ND FLOOR
- ⑩ 2" INCH FEED TO 2ND FLOOR
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- ⑫ 2" INCH FEED TO 2ND FLOOR
- ⑬ 2" INCH FEED TO 2ND FLOOR
- ⑭ 2" INCH FEED TO 2ND FLOOR
- ⑮ 2" INCH FEED TO 2ND FLOOR
- ⑯ 2" INCH FEED TO 2ND FLOOR
- ⑰ 2" INCH FEED TO 2ND FLOOR
- ⑱ 2" INCH FEED TO 2ND FLOOR
- ⑲ 2" INCH FEED TO 2ND FLOOR
- ⑳ 2" INCH FEED TO 2ND FLOOR

## PUMP SCHEDULE

PUMP	MATERIAL
P-30 — FILTER BOY TUMBLER	STAINLESS STEEL
P-31 — FILTER BOY TUMBLER	STAINLESS STEEL
P-32 — FILTER BOY TUMBLER	STAINLESS STEEL
P-33 — FILTER BOY TUMBLER	STAINLESS STEEL
P-34 — FILTER BOY TUMBLER	STAINLESS STEEL
P-35 — FILTER BOY TUMBLER	STAINLESS STEEL
P-36 — FILTER BOY TUMBLER	STAINLESS STEEL
P-37 — FILTER BOY TUMBLER	STAINLESS STEEL
P-38 — FILTER BOY TUMBLER	STAINLESS STEEL
P-39 — FILTER BOY TUMBLER	STAINLESS STEEL
P-40 — FILTER BOY TUMBLER	STAINLESS STEEL
P-41 — FILTER BOY TUMBLER	STAINLESS STEEL
P-42 — FILTER BOY TUMBLER	STAINLESS STEEL
P-43 — FILTER BOY TUMBLER	STAINLESS STEEL
P-44 — FILTER BOY TUMBLER	STAINLESS STEEL
P-45 — FILTER BOY TUMBLER	STAINLESS STEEL
P-46 — FILTER BOY TUMBLER	STAINLESS STEEL
P-47 — FILTER BOY TUMBLER	STAINLESS STEEL
P-48 — FILTER BOY TUMBLER	STAINLESS STEEL
P-49 — FILTER BOY TUMBLER	STAINLESS STEEL
P-50 — FILTER BOY TUMBLER	STAINLESS STEEL

## GEN. NOTES

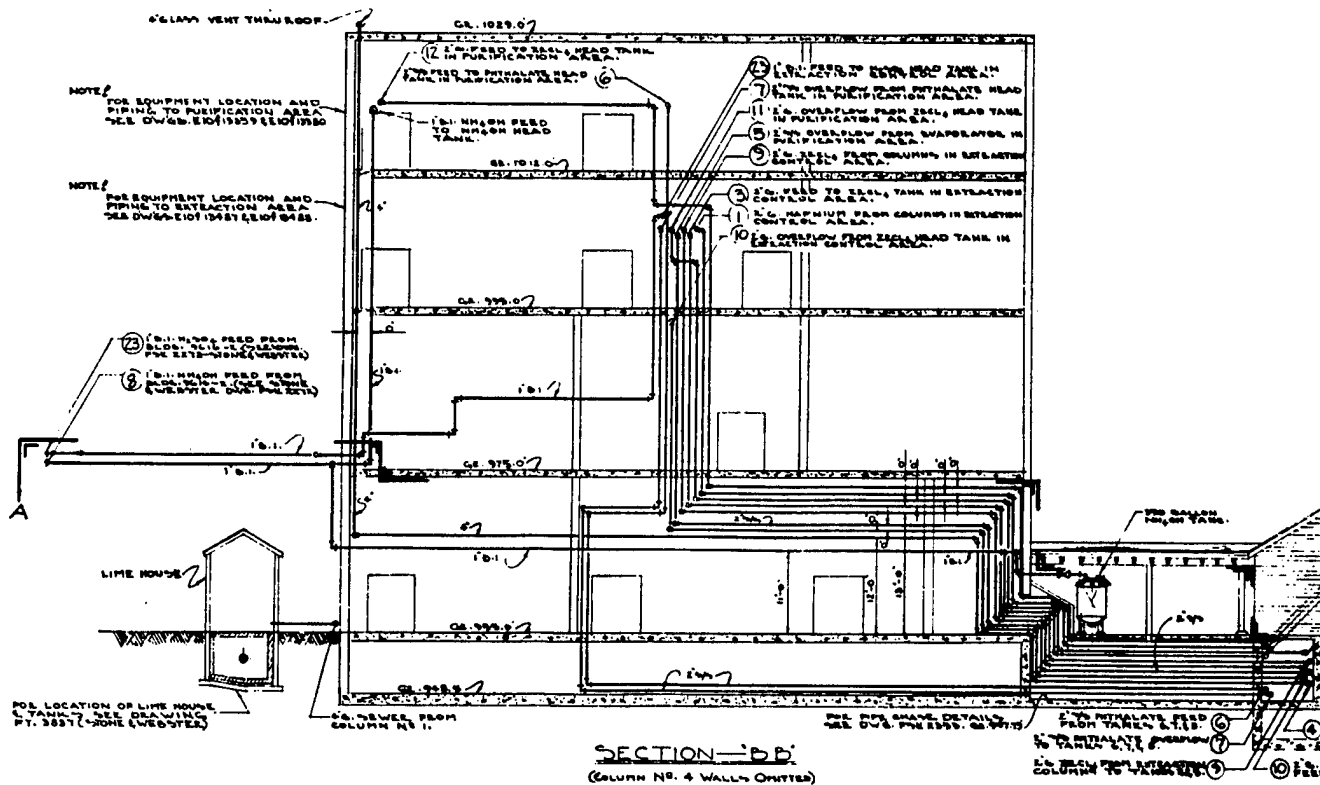
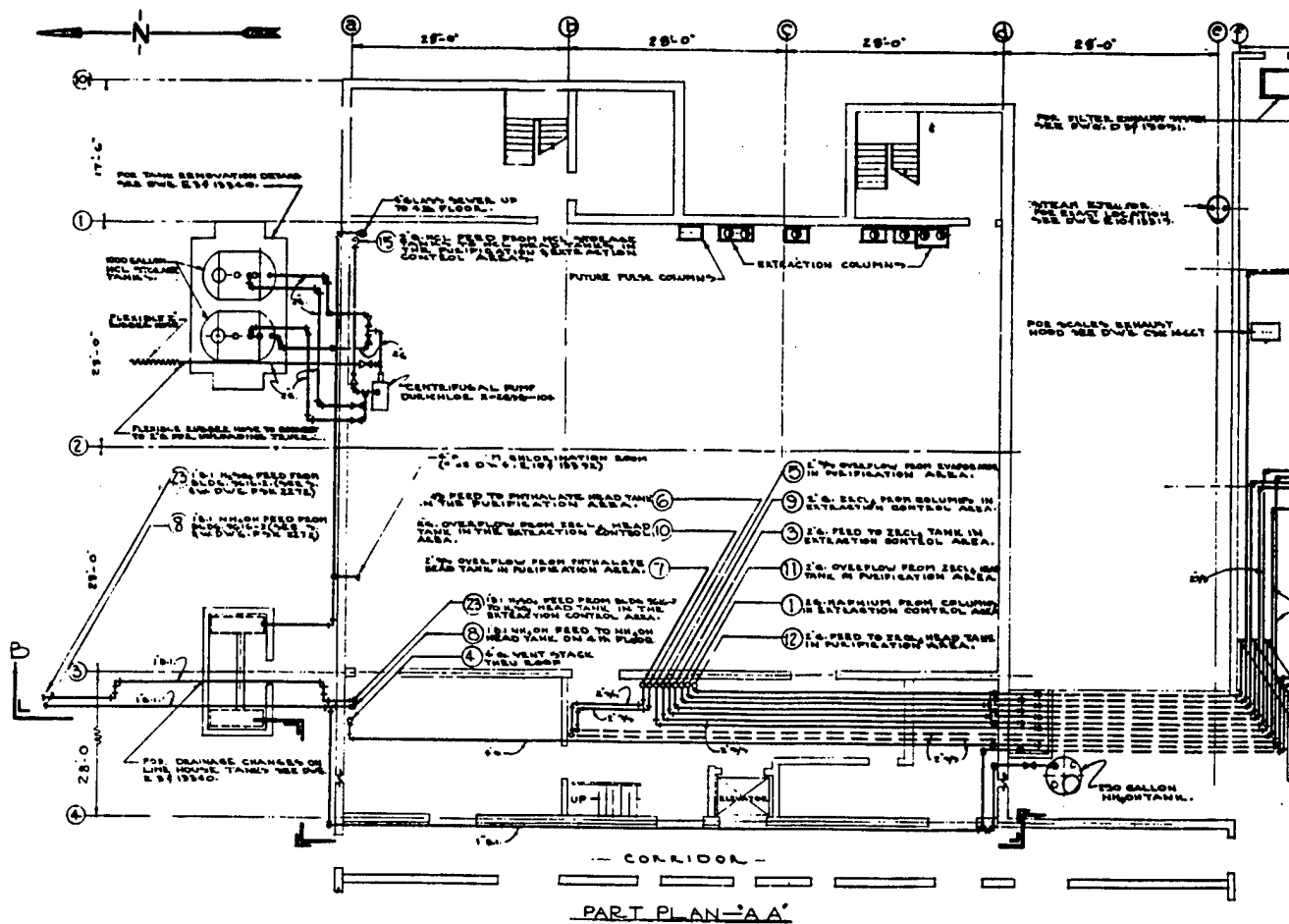
- 1. ALL GLASS PIPING SHOWN TO BE PYREX GLASS.
- 2. ALL VALVES ON GLASS LINES TO BE HILL'S CANNA GLASS LINED VALVES, EXCEPT WHERE NOTED.
- 3. USE HILL'S MECHANICAL DRAINAGE VALVES ON STEEL OR BLACK IRON LINES.
- 4. PIPING SHOWN DIAGRAMMATICALLY ONLY AND MAY BE VARYED TO MEET FIELD CONDITIONS.
- 5. PIPE SUPPORTS TO BE DESIGNED AND INSTALLED BY THE FIELD.
- 6. GLASS PIPE MAY BE SUBSTITUTED FOR BLACK IRON PIPE WHEREVER IT IS MORE EXPEDIENT.
- 7. PIPING HOOKUP FOR WOOD FILTER PRESS TO BE THE SAME AS SHOWN FOR HILL FILTER PRESS.
- 8. ALL FILTER PRESSSES TO BE EQUIPPED WITH OIL PAN, AS NOTED. OIL PAN TO BE USED AS CONTAINED IN THE FIELD.
- 9. FOR EQUIPMENT LOCATION IN THE PURIFICATION & CALCINING AREA, SEE DWG. E-101555.
- 10. DIAGRAMMATIC PUMP HOOKUP TO APPLY TO ALL FILTER BOY TUMBLER PUMPS.

## PERMANENT ZIRCONIUM PLANT PIPING LAYOUT-PURIFICATION AREA, PLAN &amp; SECTIONS

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ACKNOWLEDGEMENTS

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